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A PLASMA FLAME SPRAY HANDBOOK. (U)
MAR 77 T J ROSEBERRY, F W BOULGER

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REPORT NO. MT-043

MARCH 1977

A PLASMA FLAME SPRAY HANDBOOK

A PROJECT OF THE
MANUFACTURING TECHNOLOGY PROGRAM
NAVAL SEA SYSTEMS COMMAND

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FINAL REPORT



NAVAL ORDNANCE STATION
LOUISVILLE, KENTUCKY 40214

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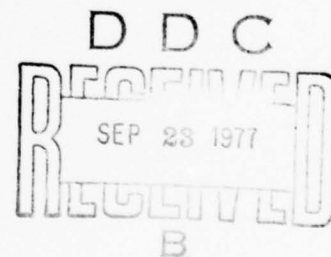
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ABSTRACT

The Plasma Spraying process is a versatile fabrication technique used to apply a wide range of coatings on various workpiece materials. Coatings are applied to restore or attain desired dimensions, to provide electrical or thermal shielding (or conduction), or to improve the resistance to abrasion, corrosion, or high temperatures. Success in meeting intended goals by Plasma Spraying depends on selecting an appropriate coating material and using cleaning and spraying techniques suitable for the materials involved. This report provides useful, up-to-date information about Plasma Spraying both metallic and nonmetallic coatings on steel and bronze. It discusses the principles and details the procedures suitable for producing high-quality coatings destined for severe service applications of interest to the U.S. Navy.

This handbook should be considered a guide, not a bible, for planners, designers, and production personnel responsible for choosing, applying, and evaluating plasma-arc sprayed coatings. Limitations on space and reliable information, and the variety of types and applications of coatings, prevented covering some aspects of plasma spraying in detail. For such reasons, differences in the types of plasma-spray systems used successfully, limiting coating thickness, and effects of specific part geometries are not covered comprehensively. Most of the information in the handbook is directed to engineers overseeing and developing expertise in plasma-arc spraying.

The handbook is based on experience, published information, and experimental data generated on this program. The experimental work was conducted with seven types of coatings deposited on the following types of workpiece materials:

Aluminum	Low-carbon steel
Aluminum bronze	316 stainless steel
K-Monel	

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FOREWORD

This is the final report of work completed under NAVORDSYSCOM Work Request WR-3-5961 issued to prepare a Plasma Spray Handbook devoted to naval ordnance requirements for use by: design engineers, production engineers, metallurgists, process planners, shop supervisors, plasma operators, and inspectors. The handbook was performed under the direction of the Naval Ordnance Station, Louisville, Kentucky, through Contract Number N-00187-73-C-4030.

The report was prepared by Battelle Columbus Laboratories and has been edited by the Naval Ordnance Station, Louisville to insure compliance and coordination with the total requirements of the NAVORDSYSCOM Work Statement.

Funding was provided by the Industrial Resources and Facilities Division (ORD-047) of Naval Ordnance Systems Command under the Manufacturing Technology Program and was completed for the Naval Sea Systems Command (SEA-070).

Acknowledgment is given to the following persons without whose help this handbook would be incomplete.

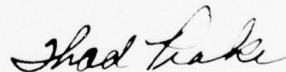
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"This Manufacturing Technology report has been reviewed and is approved."



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SECTION I

1.

INTRODUCTION

1.1

THERMAL SPRAY PROCESSES

In a thermal-spray process, a material, in the form of wire, rod, or powder, is heated to a molten or semimolten condition, atomized, propelled, and deposited on a workpiece or substrate. In most spraying processes, the material to be deposited is carried to the desired location by a stream of compressed air or inert gas. Thermal spraying techniques of various types are useful for applying a wide variety of materials on various metallic and nonmetallic substrates. Most metals, oxides, refractory compounds or cermets, and even some organic plastics, can be sprayed by using appropriate techniques and equipment. Although spray processes can be used to build up free-standing objects on fugitive or disposable substrates, this report deals only with coating applications.

Coatings are of interest to the Naval Ordnance Station because they can be used to improve the service performance of many types of components. Coatings can be applied to restore or attain desired dimensions, to provide electrical or thermal shielding (or conduction), or to improve the resistance to abrasion, corrosion, or high temperatures. This report concentrates on Plasma-Arc Spraying because it is a versatile technique well suited to producing coated components destined for severe service applications. Almost any material can be plasma sprayed. Because the operating temperatures are so high, the process is especially appropriate for applying coatings of refractory materials such as intermetallic compounds and ceramics. It is expected, however, that the most common Naval Ordnance applications will be for restoring dimensions and applying wear-resistant coatings. The information on Plasma-Arc Spraying was obtained from publications, experience, and experiments conducted on this program. The substrates used in the experiments included low alloy steel, stainless steel (Type 316) and 6061-T1 aluminum. The information is expected to be of interest to designers in choosing coatings and useful for planning and production personnel responsible for applying and evaluating coatings on specimens and end items.

This handbook emphasizes the precautions to be taken in plasma-spray processes in order to obtain high-quality coatings. The basic factors determining the quality of a coating applied by any of the common thermal spray processes are

- (1) The temperature of the material in the spray,
- (2) The velocity of the material in the spray when it impinges on the substrate, and

- (3) The severity of oxidation of the sprayed material and of the substrate during heating, spraying, and cooling.

Figure 1 shows deformed particles resulting from directing a well-heated spray onto a substrate at a suitably high velocity. The resultant coating as shown in Figure 2 displays a high theoretical density, a characteristic favoring very strong particle-to-particle coherence.

A combination of low particle velocity and minimal particle heating results in less particle deformation upon impact and consequently low particle-to-particle coherence and in voids between adjacent particles.

It follows that oxidized spray material particles will not cohere nearly as well as a clean unoxidized material.

There are seven commonly used thermal-spray processes that utilize two basic forms of energy for melting materials to be sprayed. These energy forms are gas combustion and electric arc.

Gas Combustion Heating

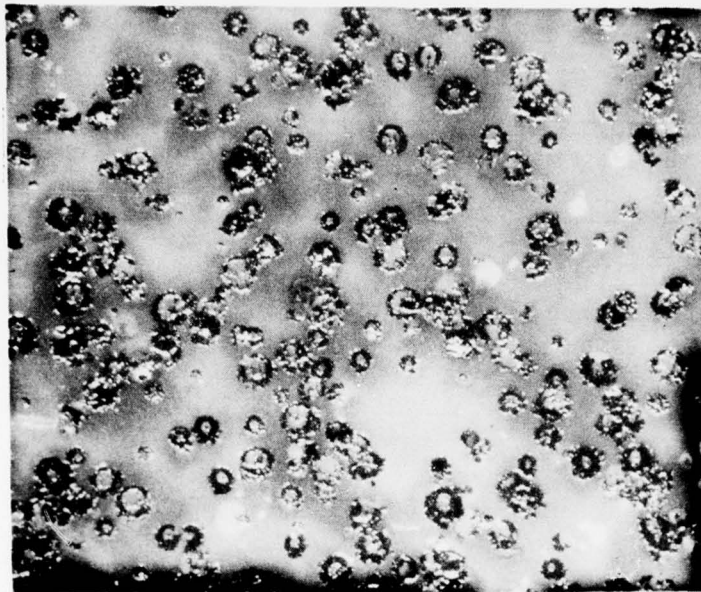
- (1) Powder Flame Spray
- (2) Wire Flame Spray
- (3) Rod Flame Spray
- (4) Detonation Spray

Electric Arc Heating

- (1) Plasma-Arc Spray
- (2) Electric-Arc Spray
- (3) Transferred-Arc Deposition

The maximum working temperature attainable differs among thermal spray methods. Furthermore, the typical particle velocities of the different methods vary in a threefold range. Both factors affect the suitability of gas-combustion and arc-heating methods for specific applications. Table I lists typical particle velocities and maximum working temperatures for the principal thermal spray methods.

Oxidation of spray particles is undesirable in the case of many metallic materials. For that reason, rapid heating and high particle velocities are desirable. Both characteristics are typical of electric-arc heating and the detonation spray processes.



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FIGURE 1. DEFORMATION OF MOLTEN OR SEMIMOLTEN PARTICLES
RESULTING FROM SPRAY IMPACTING ON A SUBSTRATE



← Tantalum
Coating

← Steel
Substrate

100X

5G085

FIGURE 2. MICROSTRUCTURE OF TYPICAL HIGH
DENSITY SPRAY COATING OF TANTALUM

TABLE I. TYPICAL PARTICLE VELOCITIES AND MAXIMUM WORKING TEMPERATURES IN THERMAL SPRAY PROCESSES

	Particle Velocity, fps	Working Temperature, F
<u>Gas Combustion Heating</u>		
Powder Flame Spray	80-120	4600-4800
Wire Flame Spray	800	5000-5200
Ceramic Rod Flame Spray		
Detonation Spray	2400	6000+
<u>Electric Arc Heating</u>		
Electric Arc Spray	800	10,000-12,000
Plasma Spray	1600	20,000+
Transferred Arc	1600	20,000+

1.1.1 Gas Combustion Heating

The only industrially common type of gas combustion process used for thermal spraying utilizes oxygen and acetylene to provide heat to the material to be deposited. The compressed gases are ordinarily supplied to the torch from storage tanks or cylinders. Acetylene (C_2H_2) burns to provide a hot flame and an atmosphere suitable for welding and spraying applications. Oxygen is supplied to support combustion. Ordinarily about 60 percent of the total oxygen comes from pressurized storage and the rest from the air surrounding the flame. The ratio of acetylene and oxygen at the tip of the torch controls the temperature and atmosphere in the flame.

A low ratio of oxygen to acetylene produces a comparatively low flame temperature and a carburizing atmosphere. Conditions of that type are used for soldering and silver brazing. Combustion of an equal ratio of gases produces a flame temperature near 5400 F and a reducing atmosphere sometimes used for melting and welding low-alloy steel. A slightly higher ratio of oxygen to acetylene, around 1.1, produces a neutral flame and slightly higher temperatures, around 5500 F, better suited to processing steel. Flame temperatures up to 6000 F and oxidizing atmospheres result from increasing the oxygen/acetylene ratio up to 1.75. That ratio can be used for welding copper and its alloys, but produces oxide slags. Oxidizing gas-combustion flames are unsuitable for welding steel or for thermal-spraying materials other than oxides.

Flames produced by the combustion of propane, natural gas, or hydrogen, with oxygen, are seldom used for thermal spray processes.

Even under highly oxidizing conditions, flame temperatures are too low, around 5000 F.

1.1.1.1 Powder Flame Spray

Torches for powder flame spraying have provisions for adding powder to the gas stream. The powder, carried by a stream of compressed air, is injected in the flame before it leaves the torch. The flow of powder is started, by operating a lever on the handle of the torch, and the material is carried by the compressed gases through the tip of the torch where the particles are heated by the flame. In most cases compressed air is used to carry the powder. The presence of powder and the propelling air stream lowers the working temperature of oxyacetylene flame sprays to approximately 4600 to 4800 F.

Deposition rates for oxyacetylene flame spraying are relatively high, compared to plasma-spray methods. This gas-combustion heating method is best suited to depositing thin coatings, on the order of 0.02 in. in thickness. The process is widely used for hard facing steel and cast iron components. The equipment for oxyacetylene flame spraying is relatively inexpensive, on the order of \$1000 for a manually operated unit.

The major shortcomings of the powder flame method result from the high proportion of heat transferred into the substrate or workpiece during spraying. The marginal available working temperatures and minimal velocities necessitate that the flame impinge on the substrate. This is most undesirable because it promotes oxidation of both substrate and coating and requires the utmost care to avoid cracking and/or spalling of the coating.

As a consequence of these conditions inherent in the flame powder-spray process, coatings produced by this process exhibit a bond, density, and overall strength that is inferior to coatings produced by other thermal-spray techniques. In view of the critical nature of performance required from Naval Ordnance hardware, it is recommended that flame powder sprayed coatings be restricted to dimensional restoration applications where wear or spalling would not result in malfunction.

1.1.1.2 Fused Powder Flame Spray

This term is used to denote a variation of the conventional flame spray practices. Certain alloys can be applied with a flame powder spray device and subsequently fused to the substrate. The treatment produces a very dense, cast structure with a metallurgical bond to the substrate. These alloys are available as either nickel- or cobalt-base materials in hardnesses ranging from Rc 20 to Rc 61. In all cases, the fusible alloys contain approximately 3 percent of boron, which acts as a flux to minimize oxidation of either the spray alloy or substrate during the fusion

treatment. Fusing may be accomplished by either torch heating, induction heating, or in furnaces with a vacuum or dry hydrogen atmosphere. The fusion temperature ranges from 1865 F to 2080 F, depending upon the type of alloy used. Care is needed in fusing to avoid distortion and contamination of coated components.

1.1.1.3 Wire Flame Spray

This thermal-spray process utilizes an oxyacetylene flame as a heat source and spray material in wire form. Figure 3 shows the components of a wire-spray facility and Figure 4 shows the cross section of a typical wire flame-spray device.

In the process, the spray material in wire form is pulled or drawn into the spray device by drive rolls that are powered by an air turbine or in some cases by an electric motor. In both cases the rate of wire feed is adjustable.

The wire is pushed into the spray nozzle where it is melted by the oxyacetylene flame. A stream of compressed air impinges on the molten tip of the wire, atomizing the molten material and propelling some to the substrate.

In the wire flame-spray process, the oxyacetylene flame is restricted to a considerably smaller diameter than in powder flame spray, which results in more effective utilization of the energy (heat) produced by the oxyacetylene flame. Consequently, higher spray rates and, in most cases, coating of superior quality may be obtained by use of the wire flame-spray process than are possible with the powder flame-spray technique. Comparative spray rates, deposit efficiencies, and coating densities are not available; the wire version is more common, especially for coatings thicker than 0.03 in.

1.1.1.4 Rod Flame Spray

In concept, this thermal-spray process is virtually identical to the wire flame-spray technique; the basic difference is that the spray material is in rod form. Figure 4 shows the arrangement used for feeding a rod into the flame. Some hard facing and other metallic materials are too brittle to be formed into wire. Such materials are fed into the torch as cast rods or sometimes as tubes filled with appropriate powders.

The rod spray process is specifically designed for the deposition of oxide coatings. For the most part, the ceramic rod-spray process has been replaced by powder flame-spray and plasma-spray techniques because of monetary and technical considerations.

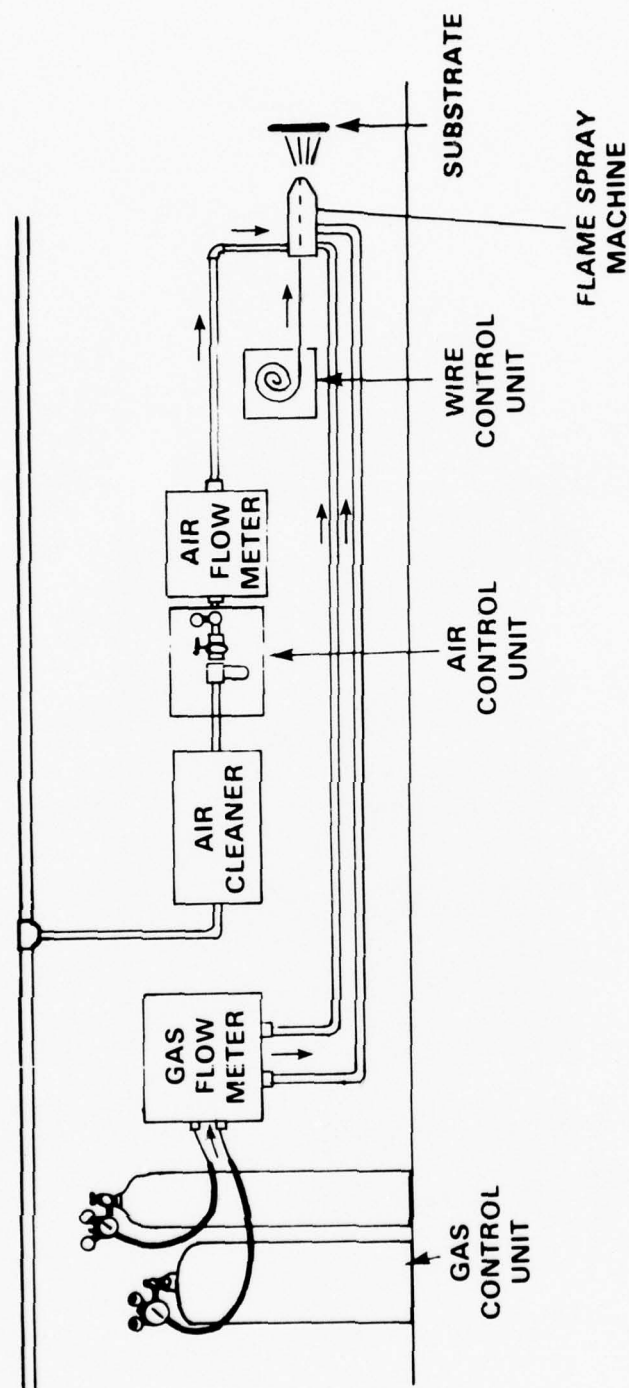


FIGURE 3. TYPICAL FLAME WIRE SPRAY SYSTEM

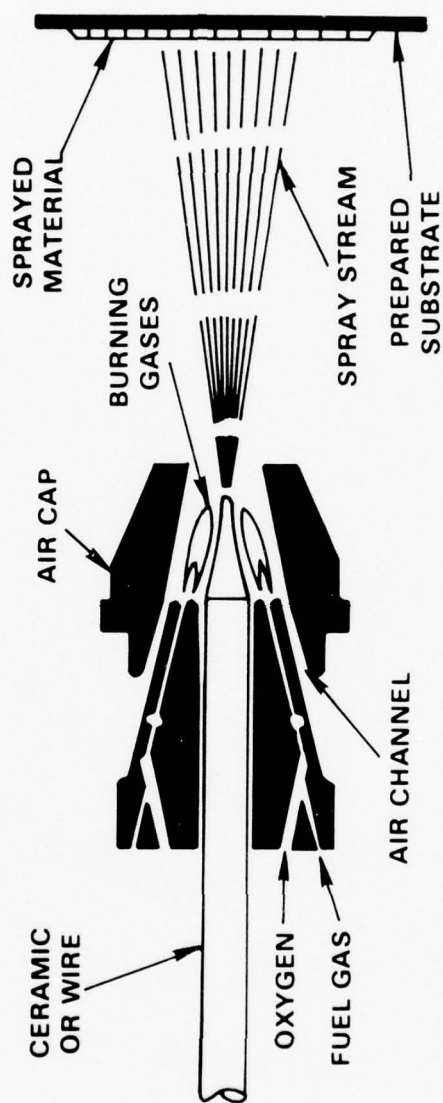


FIGURE 4. CROSS SECTION OF TYPICAL WIRE OR ROD FLAME SPRAY DEVICE

1.1.1.5 Detonation Spray

Note: It is believed that Union Carbide Corporation is the principal user and considers proprietary many of the details of the process and equipment for detonation spraying. (1)

The detonation gun or spray process utilizes the heat and velocity of shock waves created by exploding metered amounts of oxygen and acetylene in a device similar to the breech of a gun. The design and operation of the detonation gun has been described in technical and patent literature. (2,3)

The operation of a detonation spray gun can be described by reference to Figure 5. That schematic shows that the powder to be melted and sprayed is injected into the chamber where the controlled detonation takes place. The following sequence of operations takes place:

- (1) Injection of oxygen, acetylene, and powder simultaneously;
- (2) Ignition and detonation of the oxyacetylene mixture by a spark plug;
- (3) Purging the chamber with nitrogen, to prevent premature ignition of the next charge.

This sequence is repeated at a rate of eight cycles per second and is continuous until the required coating thickness has been achieved.

The spray material is heated to a molten or semimolten state as a result of being transported down the barrel of the detonation device at sonic or supersonic speeds. It is estimated that temperatures in excess of 6000 F can be generated in this manner.

The molten or semimolten particles of spray material impinge on the substrate at a velocity of approximately 2400 fps, producing a bond that may be classified as metallurgical.

The characteristically high operating temperatures and particle velocities of the detonation spray method, indicated in Table I, result in unusually high quality coatings. The higher kinetic energy of the spray particles causes more deformation on impact. The thinner particles develop a finer structure and better particle interlocking. The coatings have higher densities and stronger bonds to the substrate. Table II presents data supporting that view.

The detonation process is primarily utilized for the deposition of relatively thin (0.004-0.008 in.) wear or hard surface

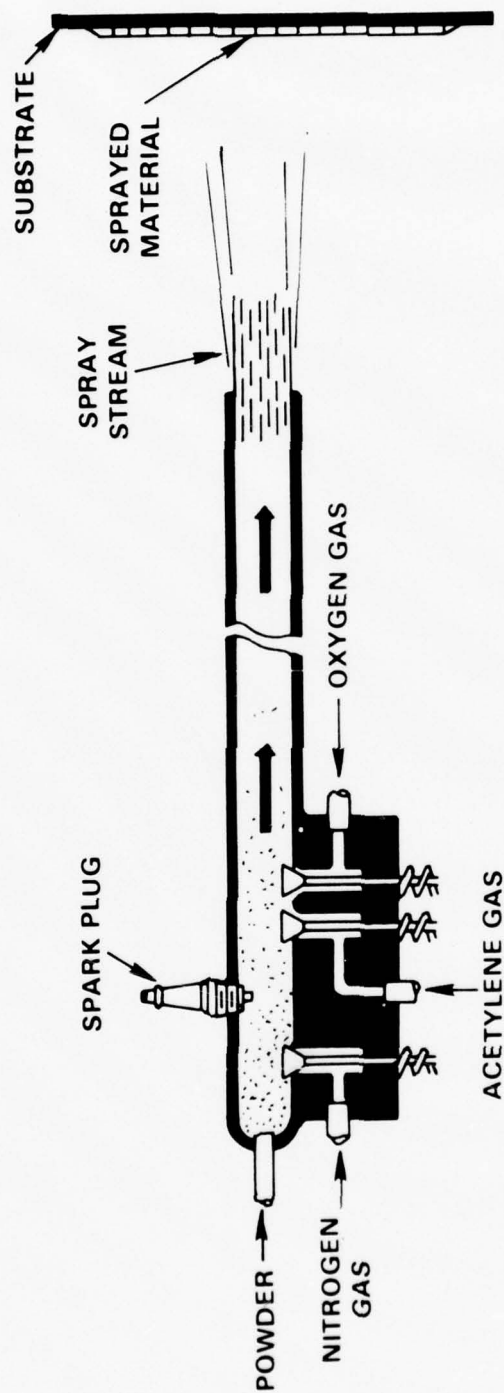


FIGURE 5. SCHEMATIC OF DETONATION SPRAY GUN

TABLE II. PROPERTIES OF COATINGS APPLIED BY DETONATION
SPRAY GUN AND PLASMA ARC METHODS (4)

Spray Method:	Gun	Plasma	Gun	Plasma
<u>Coating Composition</u>				
Tungsten carbide, percent	85	88	-	-
Cobalt, percent	15	12	-	-
Al ₂ O ₃ , minimum percent	-	-	99	99
<u>Properties</u>				
Vickers hardness	1050	750	1100	825
Bond strength(a), 10 ³ psi	>12	10	10	7.5
Rupture modulus, 10 ³ psi	100	55	20	20
Elastic modulus, 10 ⁶ psi	31	22	14	5.7
Density, g/cm ³	13.2	12.5	3.4	3.38
Porosity(b), volume percent	1	2	2	3

(a) Values determined on steel or aluminum by ASTM method C633-69.

(b) Porosity estimated from metallographic examinations.

coatings. Although the quality of a detonation coating is unsurpassed, the cost is nearly twice that of its closest process competitor (plasma spray). Because of economics, coating thicknesses of approximately 0.010 inch are the normal limits. Due to these monetary and technical consideration, the detonation coating process should be considered only in the event other thermal-spray processes prove to be inadequate.

1.1.2 Electric Arc Heating

1.1.2.1 Plasma-Arc Spray

The plasma-arc spraying method is a modification of the plasma-arc welding process. All electrical arcs ionize the gas in the conducting channel separating the two electrodes and form a plasma. The ionized form of matter known as plasma consists of free electrons, electrically neutral atoms, and positive ions. The plasma is heated by resistance to the current passing through it. Compared to gas flames, plasmas have three important advantages for heating particles to be sprayed. The plasma temperature is higher, in excess of 20,000 F, it provides better heat transfer to other objects, and a nonoxidizing atmosphere is easier to achieve.

Plasma-arc welding and spraying methods are refinements of gas tungsten-arc welding processes. In gas tungsten-arc welding

the arc is established between the tungsten electrode and the work; the unconstricted arc transfers most of the heat needed for welding. In plasma-arc processes the arc is established between the tungsten electrode and the nozzle or orifice body of the torch (Figure 6). The arc is constricted within the torch and the plasma delivers the heat to the work. The constricted arc used for plasma-arc welding confers several advantages over unconstricted arcs:

- (1) The energy is more concentrated,
- (2) Arc stability is better and dimensional control is easier, and
- (3) Plasma has a higher velocity and heat content.

The same advantages favor the plasma spray process over other approaches.

The plasma-spray process is a well developed, reliable thermal-spray process for the deposition of coatings. The inherent versatility and easy control of the process permits the deposition of a wide variety of spray materials including metals, carbides, oxides, borides, nitrides, and silicides. With proper control, the plasma-spray process is even capable of depositing organic plastics and certain glasses. In general, it may be stated that any material that does not decompose within 200 F of its melting point may be plasma sprayed. Of course, precautions must be taken to prevent the plasma from overheating and degrading the properties of the substrate.

The plasma-arc spraying process can be described by reference to the sketch in Figure 6. The working region of the spray gun is watercooled and the orifice body A, constituting the positive electrode, is made of copper. The nonconsumable electrode B, made of tungsten or of tungsten alloyed with thoria or zirconia, is the cathode. The copper anode (positive) also serves as the spray nozzle. The plasma arc is produced by direct current supplied with straight polarity from a constant-current power supply. Rectifiers having an open-circuit voltage of 80 to 160 volts and capable of supplying 50 to 1000 amperes are usually suitable.

The gun also contains channels for supplying two streams of gas. The stream of gas directed toward the tungsten electrode forms the plasma arc and leaves through the spray nozzle. That gas constricts the arc to form a stream of extremely hot plasma leaving the gun at a high velocity. The sketch shows that the temperature varies appreciably, from about 14,000 to 24,000 F, at different locations in the stream. The gas fed to form the arc-plasma must be inert and is usually rich in argon. Helium can be used, but is seldom the primary arc gas. Nitrogen is commonly used, but not when nitrogen absorption can cause embrittlement of the coating or substrate. Higher temperatures can be attained by adding 5 to 25 percent of hydrogen to the nitrogen or argon arc gas. Flow rates for the arc gas used for plasma spraying ordinarily range

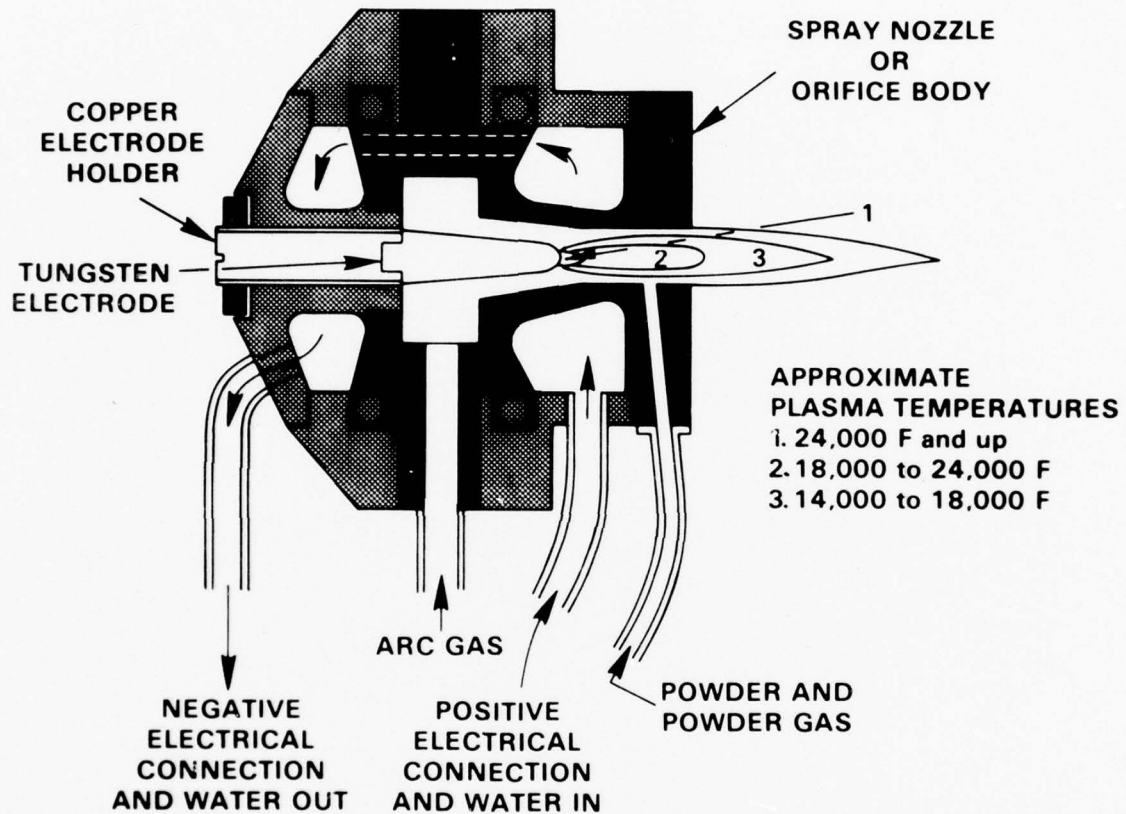


FIGURE 6. CROSS SECTION OF TYPICAL PLASMA-SPRAY GUN

from 80 to 100 cubic feet/hour (cfh). The second stream of gas, usually argon, carries the powder particles to be sprayed. Typically, the flow rates of the carrier gas range from 15 to 30 cfh.

The plasma spray system includes two other devices not shown in Figure 6. Since the plasma arc used for spraying is not struck to the workpiece, some other method of initiating a pilot arc between the two electrodes must be used. For high current applications, the arc is usually initiated by superimposing a high frequency current on the main direct current to the electrodes. The high frequency current is provided by a separate power supply. In some types of equipment the energy for initiating the arc is supplied by a charged condenser.

Plasma spray systems also contain devices for adding controlled and preselected amounts of powder to the carrier gas. The feeders are calibrated and adjustable to provide powder at the desired rates from approximately 1 to 20 pounds/hour.

The operations required for plasma spraying are:

- (1) Start flow of cooling water
- (2) Activate supply of direct current
- (3) Start flow of plasma-forming arc gas
- (4) Initiate an arc
- (5) Start injection of powder-carrying gas .

The controls of commercially available plasma spray systems are interlocked so that spraying does not start until the first four operations have been completed.

Plasma-arc powder spraying produces much higher quality coatings than the common oxyacetylene processes and can be used for a wider variety of materials. Oxygen contamination is less of a problem than it is in gas combustion processes. The plasma process is only half as expensive and more widely used than the gas detonation process. It produces coatings with densities and bond strengths approaching those of the detonation process and is applicable to thicker coatings. Plasma-sprayed coatings range from approximately 0.005 to 0.060 in. in total thickness depending on the application. The coating efficiency, the ratio between the weight sprayed and the weight deposited, ordinarily ranges from 30 to 80 percent. With careful control, the metallurgical disturbance to the substrate can be limited to about 0.005 in. The principal drawbacks of plasma-arc spraying are related to the facts that the equipment is usually not portable and that the process requires considerable skill and knowledge.

Plasma torches are usually adjusted, and nozzles chosen, to produce deposits with appropriate widths and a maximum thickness of 0.005 in./pass. Deposition rates can be varied from 1 to 20 lb/hr, depending on the materials and the coating applications.

In general, deposition rates used for metals, on a weight basis, are twice those used for non metals. Because of the high arc temperatures, the plasma torch is particularly well suited to spraying oxides and refractory ceramics. Plasma-arc and gun detonation methods are the only flame spraying methods considered acceptable for aircraft components, with the possible exception of restoring dimensions.

Table II shows that properly deposited plasma-sprayed coatings exhibit usefully high bond strengths and reasonably high values for elastic moduli and rupture moduli. Nevertheless, until recently the properties attainable by plasma spraying were not quite so good as those produced by the gun detonation process. However, the gap in capabilities between detonation gun and plasma spraying processes has narrowed. The plasma spray equipment has been improved so particle velocities are two or three times as high as those typical of older devices. The new supersonic plasma guns produce higher quality coatings and are especially desirable for spraying ceramics. The improvements affect costs. Until recently, plasma spray equipment cost on the order of \$10,000, but the new higher-velocity guns cost twice as much.

Plasma-arc spraying is ordinarily conducted in air. However, the powder-carrying gas mixes with the surrounding air and increases the oxygen content of the stream and of some metallic coating materials. The severity of oxidation of molybdenum during spraying in air has been shown to increase linearly with the distance between the gun and the substrate. Coatings with little or no oxygen contamination can be deposited in an enclosure with an inert atmosphere or by using an inert stream of shielding gas. Tucker quoted the data in Table III to illustrate the benefits of the shielding technique.⁽⁵⁾ Shrouding the spray with an inert gas apparently prevented oxygen pickup by nickel, tungsten, and titanium coatings and lowered the oxygen levels in the case of copper and molybdenum deposits.

TABLE III. OXYGEN CONTENTS OF METALLIC COATINGS PLASMA-SPRAYED IN AIR AND IN AN INERT-GAS SHIELD⁽⁵⁾

Coating Material	Oxygen Content, percent		
	Powder	Conventional Coating	Gas-Shielded Coating
Copper	0.126	0.302	0.092
Nickel	0.172	0.456	0.151
Tungsten	0.027	0.274	0.030
Molybdenum	0.419	0.71	0.160
Titanium	0.655	2.0+	0.730

Table IV indicates that densities and strengths of aluminum, stainless steel, and tungsten coatings were better for coatings sprayed in an inert atmosphere than those for coatings sprayed in air. Presumably the degradation in properties was caused by oxidation during spraying.

TABLE IV. PROPERTIES OF PLASMA-ARC SPRAYED COATINGS DEPOSITED IN DIFFERENT ATMOSPHERES (6)

Coating	Atmos- phere	Density, (a) percent	Tensile Strength, psi	Modulus of Rupture, psi
Aluminum	Argon	86	5,600	-
	Air	76	4,000	-
Stainless Steel	Argon	91	33,900	-
	Air	84	19,200	-
Tungsten	Argon	90	-	51,000
	Air	86	-	29,000

(a) Density expressed as a percentage of theoretical value.

Plasma-arc spraying in an argon-atmosphere chamber gives even better results than using a protective shroud of the same gas. Suitable spray chambers, made with double walls to permit water cooling during service, can be evacuated to pressures of 10^{-4} mm of Hg. After evacuation, the chamber is back filled with an inert gas to atmospheric pressure before spraying. Table V shows that such practices produce better bond strengths than spraying in air under a shroud of argon gas. When gas shrouding was employed, the average strength of the bond between the titanium coating and the tungsten substrate was 3940 psi. The average bond strength for specimens sprayed in a chamber filled with argon was at least 6900 psi. Since some bonds were stronger than that value, tensile failures sometimes occurred in the epoxy cement used to fasten the coated specimens to the tabs used for gripping.

Table VI shows that spraying in a chamber can even remove most of the oxygen, nitrogen, and hydrogen originally present in titanium powder material. It is generally believed that spraying in a truly protective atmosphere will not only improve the apparent density and bond strength, but also reduce the average pore size of a coating deposited under otherwise similar conditions.

TABLE V. BOND STRENGTHS OF TITANIUM-COATED TUNGSTEN SUBSTRATES PRODUCED BY PLASMA SPRAYING WITH TWO TYPES OF PROTECTIVE ATMOSPHERES

Gas-Shroud Protection		Argon Chamber Protection	
Bond Strength, psi	Type of Failure (a)	Bond Strength, psi	Type of Failure (a)
4170	I	6750	I
4180	I	6500	I
3160	RI	7280	E
4170	RI	6670	E
3740	RI	6800	E
4200	RI	7400	E
Average	3940 \pm 420		6900 \pm 360

(a) Type of Failure: I, at substrate interface
RI, random to substrate interface
E, failure in the epoxy adhesive

TABLE VI. EFFECT OF SPRAYING IN ARGON-FILLED CHAMBER ON CONTAMINANTS IN TITANIUM POWDER

Material Condition	Oxygen, ppm	Hydrogen, ppm	Nitrogen, ppm
As received	2872	630	1485
As sprayed in inert atmosphere chamber	892	357	57

1.1.2.2 Electric-Arc Spray

A common technique for electric-arc spraying of metals or other conductive materials is illustrated by Figure 7. Two wires or rods of the material to be deposited are fed through electrical contacts to form the positive and negative electrodes connected to a source of direct-current power. When the wires form a sufficiently narrow gap, an arc forms and temperatures in excess of 10,000 F are attained at the tips of the electrodes. As the consumable electrodes melt, the liquid drops are atomized and carried to the substrate by a blast of compressed air or inert gas. By using electrode wires of different compositions, this type of electric-arc spraying is capable of depositing alloy coatings.

The electric-arc spraying method is less versatile than the plasma-arc process because it is applicable only for materials with adequately high electrical conductivity. The lower working temperature and lower particle velocities result in coatings with poorer characteristics than those applied by plasma spraying. Since oxidation is less of a problem and working temperatures are higher, electric-arc spraying offers a slight advantage over flame spraying by the powder, rod, or wire gas combustion methods.

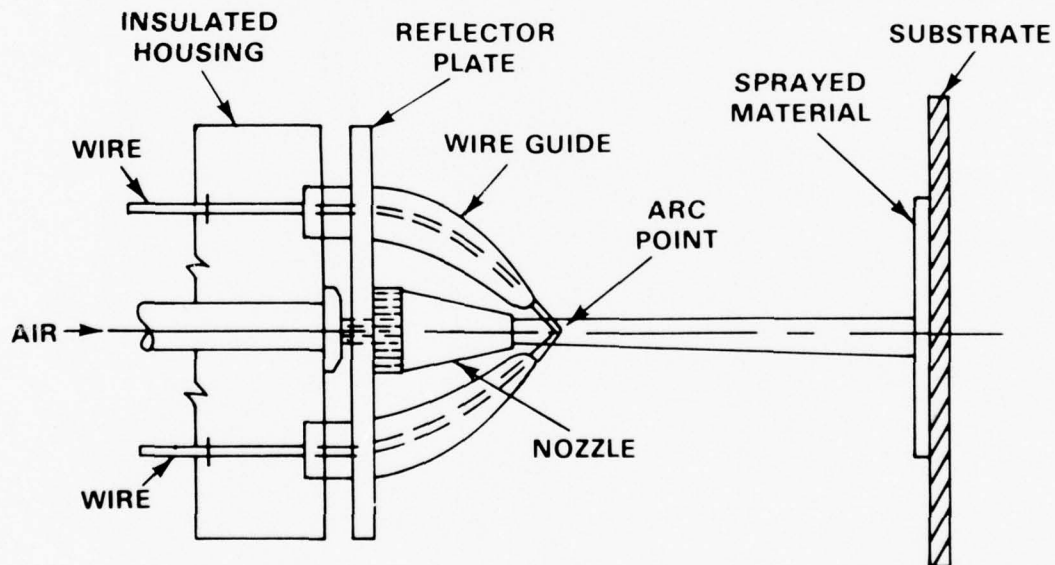


FIGURE 7. SCHEMATIC OF TYPICAL ELECTRIC ARC SPRAY DEVICE

1.1.2.3 Transferred-Arc Deposition

The transferred arc deposition process, used for depositing welded overlays, is mentioned here because it bears some similarities to plasma-arc spraying. In transferred-arc processes the discharge of electrical current occurs between the electrode and the workpiece. Since most of the heat is delivered by the arc, rather than by the plasma as in the plasma-arc spraying process, the method is called the transferred-arc deposition process.

As indicated in Figure 8, the substrate is connected to the positive side of the electrical circuit. In that respect, it differs from the plasma-arc spraying method which requires no electrical lead to the substrate or workpiece. In both cases, the tungsten-alloy nonconsumable electrode forms the negative (cathode) electrode. In the transferred-arc process, the arc to the workpiece heats and melts material near the surface of the substrate. The material needed to form the overlay is added to the molten pool of substrate material by a filler wire, or as shown in Figure 8, by sprayed powder.

Applications of the transferred-arc deposition process are limited to electrically conductive workpieces. There are other serious limitations. Since the arc extends to the workpiece to form a molten pool, great care must be taken to avoid undesirably high substrate temperatures and excessive dilution of the coating. By precise control of the arc current, the transferred-arc deposition process can deposit 0.015-in.-thick or thicker coatings while holding the depth of substrate melting and coating dilution to, respectively, 0.007 in. and 6 percent.

Fully automated versions of the transferred-arc deposition system have the following capabilities:

Width of a one-pass deposit	- 0.05 to 1-1/4 inch
Thickness of a one-pass deposit	- 0.010 to 0.25 inch
Deposition rates	- up to 15 lb/hr .

The transferred-arc process is suitable for depositing relatively heavy coatings on substrates which can tolerate some metallurgical damage and distortion. The characteristic melting of the surface material results in a smooth coating and an unusually strong bond between the deposit and the substrate.

1.1.3 Process and Equipment Comparisons

All thermal spray processes can deposit coatings with a wide range of characteristics, depending on the operating conditions employed. Therefore, the information listed in Tables VII and VIII are only rough generalizations. For instance, the rise in temperature during processing varies considerably with the mass of the substrate, coating thickness, deposition rate, materials involved, and, especially, the precautions taken to control temperatures.

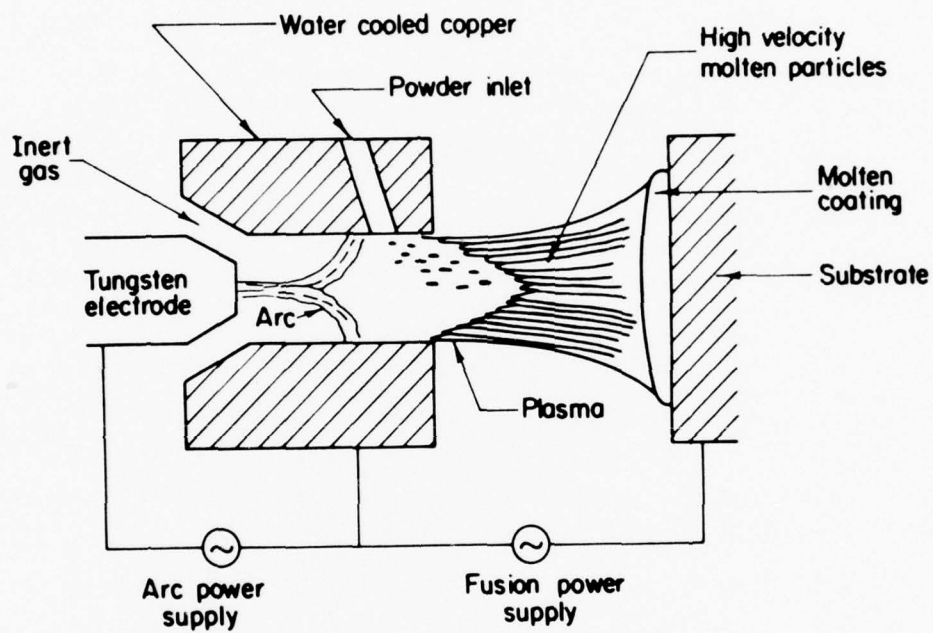


FIGURE 8. SCHEMATIC OF TRANSFERRED ARC DEPOSITION PROCESS

Table VII indicates that flame spraying equipment fueled with oxygen and acetylene is considerably less expensive than equipment based on arc or plasma heating. It also shows that the two-wire electric-arc and wire flame-spray processes are used to deposit coatings at much faster rates than the other methods.

Table VIII gives some estimates of the relative costs of depositing coatings by six thermal spray methods. The data available on the detonation-gun spray method were insufficient to include that process in the comparison. The estimates in Table VIII indicate that, for equal operating periods, the plasma-arc and transferred-arc methods are the most expensive of the processes considered.

Other factors influencing the selection of the thermal-spray process are sometimes more important than those listed in the table. Many desirable coatings have to be sprayed under conditions which will not cause contamination. The ease of doing so is a big advantage for the plasma-arc and detonation processes. The same processes are capable of higher spray temperatures and spray-particle velocities. Proper control of those features results in stronger bonds and more appropriate densities. For all of those reasons, and some mentioned earlier, Plasma Spraying and Gun Detonation methods are the only processes considered suitable for most severe service applications of interest to the U.S. Navy.

TABLE VII. COMPARISON OF THERMAL SPRAY EQUIPMENT AND PROCESSES

Equipment Type	Approximate Equipment Cost	Equipment Applications	Type of Feed Material Utilized	Type of Surface Preparation	Process Temperature, F	Particle Velocity, fps	Average Spray Rate, lb/hr
DC Plasma Arc, 40 KW Unit	\$20,000 to \$25,000	Metallic, ceramic, plastics and compounds	Powder	Grit blasting, rough threading	200-300	1600	12-15 7-20
Powder Flame Spray	\$900 to \$2800	Metallic, ceramic and fusible coatings	Powder	Grit blasting, rough threading	225-325	80-120	3-5 7-20
Wire Flame Spray	\$2100 to \$3200	Metallic coatings	Wire	Grit blasting, rough threading	175-275	800	- 5-65
Ceramic Rod Spray	\$1300 to \$3200	Ceramic and cermet coatings	Rod	Grit blasting	175-275	800	1-2 -
Two Wire Electric Arc	\$4200 to \$8400	Metallic coatings	Wire	Grit blasting, rough threading	225-325	800	- 10-120
Transferred Arc	\$20,000 to \$25,000	Metallic, fusible coatings	Powder	Light grit blasting or chemical cleaning	Fuses base metal	1600	- 7-20
Detonation Spray	Not for sale	Metallic, ceramic coatings	Powder	Grit blasting	300-500	2400	Not available

TABLE VIII. RELATIVE COSTS FOR THERMAL SPRAY METHODS (a)

	Plasma Arc		Powder		Wire		Two Wire		Ceramic	
	40 KW Unit	Arc	Flame Spray	Flame Spray	Flame Spray	Arc	Electric Arc	Transferred Arc	Rod	Spray
Original Equipment, \$	22,500	1850	2650	6300	22,500	2250				
Amortization over 5 years, \$/year	4,500	370	530	1260	4,500	450				
Maintenance at 10 percent, \$/year	2,250	185	265	630	2,250	225				
Charge per year, \$	6,750	555	795	1890	6,750	675				
Charge rate, \$/hr (b)	6.75	0.56	0.80	1.89	6.75	0.68				
Operating cost, per hour										
Labor and Overhead at \$12/hr, \$	12.00	12.00	12.00	12.00	12.00	12.00				
Oxygen (0.015¢/cf), \$	-	0.89	1.08	-	-	1.08				
Acetylene (0.056¢/cf), \$	-	2.15	3.20	-	-	3.20				
Argon (0.055¢/cf), \$	8.25	-	-	-	-	5.50				
Hydrogen (0.028¢/cf), \$	0.14	-	-	-	-	-				
Nitrogen (0.02¢/cf), \$	-	0.40	-	-	-	-				
Compressed air, \$	-	-	0.75	1.60	-	0.75				
Electricity (0.0375¢/kwh)	1.43	-	-	0.75	-	0.56				
Water, \$	0.11	-	-	-	-	0.11				
Operating Cost, \$/hr	21.93	15.44	17.03	14.35	18.17	17.03				
Total, \$/hr	28.68	16.00	17.83	16.24	24.92	17.71				
Relative Cost, \$/hr	1.6	0.9	1.0	0.9	1.4	1.0				

(a) Costs of surface preparation, jigs, fixtures, inspection, and post treatments are not included.

(b) Based on 50 percent utilization of equipment for one 8-hour shift per day, 5-day week, 50 weeks/year, or 1000 hours/year.

SECTION II

2. COATING DESIGN CRITERIA FOR ENGINEERING APPLICATIONS

2.1 COATING SELECTION

The selection of a coating for a specific end use is not, as it may seem, a matter of simply choosing a coating material with certain desired properties, for application to base material. Rather, the selection of the coating material should be approached as a design problem, taking into account such factors as the function the coating is to perform, the conditions under which it will be operating, and the physical and chemical properties of both the coating material and the base material. Careful evaluation of these factors may indicate the need for a graded or composite coating and will have a bearing on the coating application technique to be used. Final selection of the coating must be based on careful testing and evaluation of a suitable number of test specimens.

The selection of a coating for a new application is far more complex than selecting structural materials produced and fabricated by more conventional processes. In those cases, the physical and mechanical properties of the work piece material are well enough established that service performance is reasonably predictable. This is far less true of thermally sprayed coatings because the process affects the characteristics of the material deposited. Probably because of the difficulty of testing, relatively little information on properties of sprayed coatings is available. It is universally accepted, however, that the properties of sprayed coatings may be appreciably different from those expected of the powder-feed material in the virgin condition and tested in larger sections.

The properties of the deposit can be affected by various parameters of the spraying process. Kuijpers and Zaat, for example, showed that molybdenum will absorb some oxygen and nitrogen during plasma spraying, with argon arc gas, in an air atmosphere.⁽⁷⁾ In their experiments, the oxygen content of the deposit increased from 0.5 percent to 2.5 percent as the nozzle-to-substrate distance increased from 2 to 8 in. Because the spray particles are quenched so quickly by contact with the cold substrate, the microstructural characteristics are quite different from bulky specimens of molybdenum. The compositional ranges for homogeneous solid solutions are much wider and the grain sizes are smaller. Those effects influence microhardness and can be appreciable. Microhardness measurements showed that the hardness of the molybdenum coating varied from approximately 660 Vickers at the bond interface to 360 Vickers at a distance of 0.007 in. from the substrate. Similar variations in microhardness of the plasma-sprayed molybdenum coatings were found to be associated with differences in the thermo-physical properties of the substrate. For example, faster quenching

of molybdenum droplets associated with thinner deposits on a silver substrate resulted in twice the hardness of coatings on alumina substrates.

For such reasons and because of the critical nature of Naval Ordnance Equipment, it is recommended that performance in full scale or simulative tests be used as the primary criterion for validating the selection of a thermally sprayed coating for a specific application. That recommendation is not unusual; it resembles the practice commonly followed for novel processes, components, or equipment before using them in critical applications. That practice, of full-scale coating evaluation, is followed by aircraft engine manufacturers, the main users of plasma-sprayed coatings.

Regardless of the specific function a coating is intended to perform, the total operating environment should be considered when choosing a candidate material. For instance, though the primary function of a particular coating may be wear protection, it must be considered whether that coating will be subjected to excessively high (or low) temperatures or whether it will be exposed to any corrosive media. In selecting materials for thermal protection, questions of corrosive environments and exposure to abrasion or impact must be addressed. Definition of the total operating environment is essential for successful coating performance.

Selection of a coating material must be based not only on its properties as related to its end use and service environment, but on factors related to its compatibility with the substrate as well. Probably the most important such factor is the relationship between the respective coefficients of thermal expansion (CTE) of the coating material and the substrate. Larger differences in thermal coefficients of expansion cause higher stresses between the substrate and coating. Although they can be minimized by proper preheating or temperature control, and by using bonding coats with intermediate properties, it is good practice to employ coatings with thermal-expansion coefficients close to that of the substrate if other characteristics are approximately equal.

2.1.1 Coating Functions

The versatility of the plasma-arc spraying process permits it to be used for depositing over 200 different materials. Coatings of metals, alloys, oxides, and other compounds, either singly or in combination, have been used to solve many material problems. Some of the applications are suggested in Table IX and types of coatings used for specific purposes are identified in Table X.

TABLE IX. SOME APPLICATIONS OF PLASMA-ARC DEPOSITS

Anti-Wetting (stop offs)	Nuclear Moderator
Catalyst	Nuclear Shielding
Corrosion Resistance	Oxidation Protection
Electrical Conductivity	Parting Agents
Electrical Resistance	Salvage
Electromagnetic Shielding	Thermal Barrier
Emissivity	Wear Erosion
Fabrication of Freestanding Shapes	Wear Fretting
	Wear Friction

For Naval Ordnance application, plasma-sprayed coatings are of interest mainly for the following purposes: (1) thermal protection, (2) wear resistance, (3) corrosion protection, (4) electrical insulation or conduction, or (5) salvage. Some materials have suitable properties to permit use in several of these classifications. However, there are instances where a combination of materials must be used to achieve a particular property. The design of a coating for a given application should begin with the consideration of those materials whose properties are known to be useful within the desired range of temperature, hardness, electrical resistivity, etc. It is then necessary to take into account those factors, such as thermal expansion or conductivity, susceptibility to oxidation or reduction, electrical properties, etc., which would make them unsuitable for the application at hand.

2.1.1.1 Thermal Barrier

The prime requisite for a thermal barrier coating is a high melting point. This is not, however, the only requirement. Low thermal conductivity and high resistance to thermal shock are needed as well. Zirconium oxide and alumina are probably the most commonly used thermal barrier materials, though this class of materials could include most of the refractory metal oxides.

The approximate melting points of some thermal insulators and metal oxides are listed in Table XI. Table XII lists the coefficients of thermal expansion of some materials that may be of interest as substrates or bond coats. The table also lists thermal-conductivity coefficients for bulk (solid) samples of metals, but none were found for thermal barriers except the one shown for magnesia. There is little doubt, however, that the oxides have thermal conductivity values much lower than any of the metals.

TABLE X. THERMAL SPRAY COATINGS APPLICATION AND USE

Service Requirement	Coating Material	Deposition Method*
<u>Bond Coatings</u>	Nickel Aluminum (95:5)	P/A; P-F/S
	Nickel Aluminum (80:20)	P/A; P-F/S
	Nickel Aluminum (83:17)	W-F/S
	Molybdenum	P/A; W-F/S
<u>Clearance Control</u>		
<u>Abradable</u>	Nickel Graphite (85:15)	P-F/S
	Nickel Graphite (75:25)	P-F/S
	Aluminum (99.0%+)	P/A
	Silicon Aluminum (12Si)	P/A
	Silver Copper Zinc Alloy	W/F/S
<u>Abrasive</u>	Aluminum Titania	P/A
	Cermet (Al_2O_3 + NiAl)	P/A
<u>Salvage and Repair</u>	Nickel Chromium	P/A; W-F/S
	Nickel Chromium Aluminum	P/A; P-F/S
	Nickel Aluminum	P/A; P-F/S
<u>Environmental</u>	FeCr Alloy	P/A
	Zirconia	P/A
	Aluminum	E/A
<u>Instrumentation</u>	Nickel Chromium Alloy	W-F/S
	Nickel Aluminum (83:17)	W-F/S
	Nickel Aluminum (95:5)	P/A
	Nickel Chromium Silicon Boron Alloy	P/A
	Alumina	R-F/S
<u>Fretting and Wear Protection</u>	Tungsten Carbide + Cobalt (88:12)	P/A
	Tungsten Carbide + Cobalt (85:15)	D-Gun
	Titanium Carbide	P/A
	Chromium Carbide + Nichrome	P/A
	Molybdenum	W-F/S; P/A
	Copper Nickel Alloy	P/A
	Copper Nickel Indium Alloy	P/A
	Nickel Silicon Boron Alloy	P/A
	Chromium Silicon Molybdenum Cobalt Alloy	P/A
	Cobalt Chromium Nickel Tungsten Alloy	P/A
<u>Special Purpose</u>		
<u>Sacrificial</u>	Molybdenum	W-F/S; P/A
<u>Component Balance</u>	Nickel Aluminum (95:5)	P/A
<u>Speed Sensors</u>	Nickel Iron Alloy (Mu Metal)	P/A

* P/A - Plasma Arc; W-F/S - Wire-Flame Spray; P-F/S - Powder-Flame Spray; R-F/S - Rod-Flame Spray; E/A - Electric Arc; D-Gun - Detonation Flame Spray.

TABLE XI. APPROXIMATE MELTING POINTS, IN DEGREES F, OF SOME MATERIALS THAT CAN BE DEPOSITED BY PLASMA SPRAYING (8)

<u>Metals</u>			
Aluminum	1220 F	Nickel	2650 F
Aluminum bronze	1940 F	Molybdenum	4750 F
Boron	4170 F	Palladium	2830 F
Cadmium	610 F	Platinum	3225 F
Chromium	3435 F	Steel, alloy	2550/2800 F
Columbium	4530 F	Steel, stainless	2550/2750 F
Cobalt	2725 F	Tantalum	5480 F
Copper	1980 F	Tungsten	6100 F
Iron	2800 F		
<u>Oxides</u>			
Alumina (Al_2O_3)	3660 F	Silica (SiO_2)	3110 F
Beryllia (BeO)	4585 F	Tantalum oxide (Ta_2O_5)	5595 F (d)*
Ceria (CeO_2)	4710 F	Thoria (ThO_2)	5520 F
Chromium oxide (Cr_2O_3)	3615 F	Titanium oxide (TiO)	3180 F
Hafnium oxide (HfO_2)	5090 F	Titanium oxide (Ti_2O_3)	3865 F (d)
Magnesia (MgO)	5070 F	Yttria (Y_2O_3)	4370 F
Nickel oxide (NiO)	3795 F	Zirconia (ZrO_2)	4920 F
<u>Borides, Nitrides, Carbides</u>			
Boron nitride (BN)	5430 F (s)*	Boron carbide (B_4C)	4440 F
Silicon nitride (Si_3N_4)	3450 F (s)	Chromium carbide (Cr_3C_2)	3435 F
Chromium boride (CrB)	5000 F (?)	Hafnium carbide (HfC)	7025 F
Tantalum boride (TaB)	5430 F (?)	Molybdenum carbide (MoC)	4875 F
Titanium boride (TiB_2)	4710 F (?)	Molybdenum carbide (Mo_2C)	4675 F
Zirconium boride (ZrB_2)	5400 F	Tantalum carbide (TaC)	7015 F
		Tungsten carbide (WC)	5200 F
		Vanadium carbide (VC)	5090 F
		Zirconium carbide (CrC)	6400 F

* (d) - decomposes; (s) - sublimes.

TABLE XII. THERMAL AND ELECTRICAL CHARACTERISTICS OF SOME METALS MEASURED ON SOLID SPECIMENS AT TEMPERATURES BETWEEN 65 AND 210 F(8)

Material	Electrical Resistance (a)	Thermal Conductivity (b)	Coefficient of Thermal Expansion (c)
Aluminum	2.8	0.57	23.8
Aluminum bronze	12.0	0.18	17.0
Brass, yellow	6.4	0.28	19.0
Copper (99.5%)	1.7	0.93	16.8
Iron	9.7	0.20	12.3
Magnesia (d)	-	0.05	-
Manganese bronze	7.2	-	22.0
Molybdenum	5.2	0.34	4.9
Nickel	9.5	0.14	14.0
Steel, carbon	14.0	0.11	13.0
Steel, stainless	74.0	0.35	6.0
Tantalum	12.5	0.13	6.7
Titanium (99%)	58.0	0.45	9.0
Tungsten	8.0	0.38	4.6

(a) Electrical resistance in units of microhms-cm.

(b) Thermal conductivity in units of cal/sec.cm.°C.

(c) The unit for coefficient of thermal expansion is the ratio of the change in length per °C (1.8°F) to the length at 0 C (32 F) multiplied by one million.

(d) The thermal conductivities of magnesia and alumina are similar and about twice the values for CeO and ThO₂.

2.1.1.2 Wear Resistance

Wear-resistant coatings are most often required to withstand the effects of abrasion. This abrasive wear is usually accompanied by elevated temperatures, either resulting from friction or associated with the operating environment. Often, wear-resistant coatings are required for applications where impact wear is encountered or in corrosive environments. Consequently, wear-resistant coatings, as a class, must be hard, though not brittle, and possess considerable thermal- and chemical-resistant qualities. Materials with this combination of properties are generally found among the carbides and the nickel or cobalt-base alloys. Some such materials are listed in Table X and as cermets in Table XIII.

TABLE XIII. SOME MATERIALS THAT CAN BE DEPOSITED
BY THE PLASMA-SPRAY PROCESS (10)

Cermets	Plastics
Aluminum oxide - cobalt	Chlorinated polyether (Penton)
Aluminum oxide - nickel	Epoxies
Aluminum oxide - nickel aluminide	Nylon
Magnesia - nickel	Polyoxymethylene (Delrin)
Titanium diboride - chromium	Polytetrafluoroethylene (Teflon)
Titanium diboride - nickel	
Zirconium diboride - chromium	
Zirconium diboride - nickel	
Zirconia - nickel	
Zirconia - nickel aluminide	

Among others, Roseberry, Onesto, and Dufrane studied the friction and wear performance of plasma-sprayed coatings and bushing materials.⁽⁹⁾ They found that the plasma-coated components could be used in hydraulic cylinders to obtain performances expected by using conventional materials of similar design in the same mode of operation. Table XIV is a summary of their friction and wear data. The authors urged that the findings indicated in the table be treated with caution in trying to interpret them for other sliding applications for the following reasons:

1. The results were obtained from complete hydraulic cylinders, which prevents a rigorous measurement of friction and wear because of the possible influences of misalignment and variations in geometry from component to component in the various cylinders.
2. Wear performance is strongly influenced by the presence of lubricants. The tests were all conducted using MIL-F-17111 hydraulic fluid as the lubricant. The use of an alternative lubricant may alter the performance of the various combinations, while operation with no lubricant would probably invalidate the results completely.
3. The extension of any wear data from one sliding application to another is valid only if the same wear mechanism is maintained. Dramatic performance differences are experienced when the combination of sliding speed, contact pressure, atmosphere, and temperature result in operation in a different wear regime.

TABLE XIV. PERFORMANCE SUMMARY OF CYLINDER ROD COATINGS AND BUSHING MATERIALS FOR USE IN HYDRAULIC CYLINDERS (9)

Cylinder Rod Coating	Sintered Iron	Steel	K-Monel	Aluminum	Stainless Steel	Aluminum
	(Conventional)	(1020)	(Monel 500)	Bronze	(Type 316)	(6061)
$\text{Al}_2\text{O}_3\text{-TiO}_2$	A	C	C	A	A	B2
Cr_2O_3	A	A	A	B2	C	B2
Nichrome	B1	C	C	C	C	C
Molybdenum	C	B1,2	C	B1,2	B1,2	C

Key: A - Recommended combination
 B - Not recommended because of (1) high friction, and/or (2) high bushing wear
 C - Not evaluated.

Other conclusions based on the friction and wear results are as follows:

1. The wear of the plasma-sprayed coatings was negligible in all cases, which is similar to the results obtained with the conventional steel shafts.
2. The two ceramic plasma-sprayed coatings resulted in superior friction and wear performance compared with the two metallic coatings.
3. Bonding between the plasma-sprayed coatings and the various shaft materials was satisfactory; no instances of blistering or peeling were observed. (Spraying parameters are described in data sheets included in this handbook.)
4. Aluminum and stainless steel bushings experience high wear rates and appear unacceptable in a cylinder-bushing application.
5. The plasma-sprayed coatings were unaffected by the ASTM salt-fog test.

The following conclusion was drawn from the results regarding the performance of organic fillers to seal the porosity in the plasma-sprayed coatings:

Loctite 290 sealer, a low-viscosity, single-component polyester-type resin which penetrated open porosity by means of capillary action, acts as an effective sealant in both hydraulic oil and air applications. Surface porosity

observed after grinding is apparently due to the exposure of totally closed pores, which no sealant will penetrate. A post-finishing treatment with a surface sealant, such as Epon 828 epoxy provides an improved surface.

Van Wyk conducted preliminary friction and wear studies of plasma-sprayed coatings for bearings.⁽¹¹⁾ The laboratory screening tests were made with a silicon-nitride slider in contact with various ceramic coatings on titanium. Those studies and tests on plain spherical bearings showed some promise, but it was concluded that further development is required.

Truskov concluded that properly applied coatings of bronze, sprayed in an argon atmosphere, are suitable for some anti-friction applications.⁽¹²⁾ Nevertheless, the bond strengths he reported, in the range of 1300 to 2000 psi, seem low.

Levy and Morossi investigated the fatigue and erosion resistance of hard-faced titanium alloys.⁽¹³⁾ Erosion tests with sand ranked the coatings in the following order of decreasing wear resistance:

- Titanium carbonitride, over a nickel bonding coat
- Boron carbide
- Chromium overlay on a diffusion-bonded nickel undercoat
- Plasma-sprayed boron .

All of the specimens had fatigue strengths 20 to 80 percent lower than the untreated titanium alloy, but shot peening was said to confer some benefits.

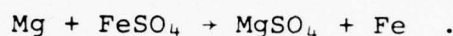
In 1975, Preece and his coworkers started a fundamental study on the mechanisms of the deterioration of plasma-sprayed coatings by erosion and corrosion.⁽¹⁴⁾ Most of the effort since then has been concentrated on two simple coatings, alumina and aluminum, sprayed on steel substrates. Although some cavitation tests were made, data are scanty because the effort was concentrated on characterization of coatings.

2.1.1.3 Corrosion Protection

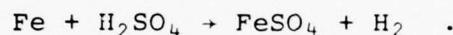
Selection of materials for corrosion protective coatings is probably the most complex coating design task. The combinations of temperature, environment, and the various modes of corrosion mechanisms possible under a given set of operating conditions impose stringent requirements on candidate materials. Corrosive environments may be either aqueous or gaseous and can span a wide range of temperatures. In some cases, gaseous products such as exhaust fumes or uncombusted fuel gases react with water under the proper temperature conditions (e.g., marine turbines) to provide a very complex corrosive environment. Basically, there are three classes of corrosion protection mechanisms. These are (a) exclusion of the corrosive environment, (b) electrolytic

protection, and (c) inhibition of corrosive action. The first mechanism is dependent on producing a coating that is impermeable to the corrosive medium and does not react with it. The second corrosion-protecting mechanism makes use of the properties of the base material and candidate coating material and requires a knowledge of their relative positions in the electromotive series. Choice of the proper coating material will determine whether the coating is anodic or cathodic to the base material and to what degree. An improper coating-substrate combination can result in a galvanic effect which could aggravate the corrosion process.

Table XV gives the electromotive series for some pure elements. It may be a rough guide even though the potentials vary for alloys of the elements. In general, any metal will react with a salt to replace a metal below it in the series, e.g.,



Metals above hydrogen in the table will react with HCl or dilute H_2SO_4 to replace the hydrogen, e.g.,



The voltage of a galvanic couple depends on the difference between the standard electrode potentials of the metals involved. For example, a zinc-copper couple generates a larger electromotive force than the iron-copper couple. In general, the electromotive series seems to fit the simple theories of rusting or oxidation of metals in air and of reactions with steam to liberate hydrogen.

Some protection can also be obtained by using a chemical corrosion inhibitor as a sealer for a sprayed coating. For this approach, the porous sprayed coating is impregnated with a corrosion-inhibiting primer or filled with an inorganic sealer. This may permit the use of a thinner coating than would be needed to exclude completely the corrosive environment. The choice of a corrosion-protective coating material must be made in light of information about the conditions the coating will have to endure. In some instances a corrosion-resistant metal may be applied, in others, perhaps a ceramic oxide may be required.

Calabrese and Coda demonstrated the importance of preventing substrate corrosion under hard coatings used in aggressive environments.⁽¹⁵⁾ They found that corrosion occurred in the substrate, during storage for several months, after a shaft had been tested as a component in steam-lubricated bearings. The substrate contained chromium and carbides. The corrosion was attributed to coating porosity sufficient to trap some of the water condensate. A short investigation on twelve types of coated specimens supported that explanation. The study also showed that corrosion could be minimized by using a corrosion-resistant, dense bond coating adherent to both the substrate and the hard coating.

TABLE XV. PARTIAL SERIES OF THE ELEMENTS ARRANGED
IN ORDER OF THEIR ELECTROMOTIVE FORCES (a) (16)

Element	Ion	Electrode Potential
Magnesium	++	+2.40
Aluminum	+++	+1.70
Manganese	++	+1.10
Zinc	++	+0.76
Chromium	++	+0.56
Iron	++	+0.44
Cobalt	++	+0.28
Nickel	++	+0.23
Iron	+++	+0.05
Hydrogen	+	0.00
Copper	++	-0.34
Copper	+	-0.47
Palladium	++	-0.82
Platinum		-0.86
Gold	+	-1.5

(a) Standard electrode potentials at 77 F.

2.1.1.4 Electrical Insulation or Conduction

Plasma-sprayed coatings are sometimes used as electrical conductors; printed circuits and contacts for certain types of furnace heating elements are examples. Coatings of oxides or organic plastics are sometimes applied to provide electrical insulation or as dielectrics. Although the electrical properties of the materials may be affected by plasma spraying, it is common practice to choose the materials on the basis of their known properties and service conditions. Then the success of the plasma-sprayed insulators or conductors is verified before the material and process conditions are adopted for production.

2.1.1.5 Salvage

Plasma spraying is often used to restore the dimensions specified for a particular component. The salvage operation may be desirable for parts inadvertently machined to the wrong dimensions or for parts worn or corroded in service. In the first type of application, the material to be deposited by flame spraying is usually identical or similar to the base material. Corroded or worn parts are usually machined to a suitably smooth finish, grit blasted, and then coated. Depending on service requirements, the coatings may be of the wear-resistant, corrosion-resistant, or friction-reducing types, or similar to the material in the substrate.

SECTION III

3. SURFACE PREPARATION

3.1 GENERAL CONSIDERATIONS

This section of the Handbook is important because over half the failures in thermal sprayed coatings is traceable to improper surface preparation. Regardless of the type of intended use of a plasma-sprayed deposit, a coating is effective only as long as it remains firmly attached to the base material. The quality of adherence, or the bond between the coating and substrate, is directly related to the cleanliness and roughness of the base material and to its chemical affinity to the coating. A thorough understanding of, and careful attention to, the details involved in preparing a surface to accept a plasma-sprayed coating will enhance the chances for successful coating. In order to appreciate the necessity for proper surface preparation, one should be familiar with the mechanics of coating-to-substrate adhesion and the factors by which it is influenced.

3.1.1 Bonding Mechanisms

There is no universally acceptable description of the mechanism of bonding between a substrate and a thermal-spray coating.

One school of thought holds that the bond is exclusively or mainly mechanical and results from sprayed particles interlocking with the roughened surface of the substrate or the peaks and valleys of previously sprayed coating material. The importance of mechanical bonding is supported by experience that surface roughening is usually necessary for achieving useful bonds between most plasma-sprayed coatings and substrates.

The main alternative explanation is that bonding results primarily from solid-state reactions. That is, a small degree of diffusion occurs between the sprayed particles and the extremely thin peaks on the roughened substrate. The existence and quality of that type of bond depend on some mutual solubility of the coating and substrate materials. Even for the most favorable conditions, however, solidification and cooling times are too short to allow diffusion reactions to form a measurably thick metallurgical bond.

Van der Waals forces, which cause mutual attraction and cohesion between two clean surfaces in close contact, are also believed to provide some bond strength under certain conditions. That type of bonding accounts for the adhesion of the "silver"

layer on glass mirrors with smooth surfaces. Regardless of the bonding mechanism, an appropriate surface texture should improve bond strength, because the true surface area is always larger than the apparent (projected) surface area.

With the current state of knowledge, it seems safest to assume that coating-to-substrate bonding results from a combination of physical, mechanical, and chemical factors. Presumably the relative importance of the various bonding mechanisms varies for different combinations of coatings and substrates and with processing parameters.

3.1.2 Coating Stresses

Stresses high enough to crack the substrate can form from rapid heating produced by the impact of plasma-sprayed particles, or in subsequent rapid cooling. In spraying zirconia on tungsten bond coats, the cracks apparently form during the instant of spraying, because the cracks are sometimes filled with the coating material.

However, the biggest factor working against good coating adherence is the stress produced by shrinkage of the sprayed coating as it cools. These stresses increase as the coating thickness is increased, putting the coating under increasing tension and ultimately resulting in the coating cracking or breaking free of the substrate. The differences in thermal contraction and expansion that cause the stresses can be reduced to some extent by substrate temperature control, but are best dealt with by proper substrate preparation, which serves to localize the stresses and limit shrinkage strains. In some cases proper temperature control results in stresses that can measurably improve the apparent bond strength. This is most likely to happen in coatings deposited on cylindrical rods or shafts.

3.2 CLEANING

Attaining a suitable bond depends, among other factors, on an intimate contact between the sprayed particles and a clean substrate with an adequately roughened surface. The first step in preparing a workpiece for thermal spraying is to remove all surface contaminants such as scale, grease, and paint.

Vapor degreasing is the most economical and efficient method of removing most surface contaminants such as greases, dust, and body oils deposited during handling. In some cases, vapor degreasing is precluded because the part is too large or is attached to another component, e.g., armature with windings. In such cases, the area to be coated should be cleaned by hand with a solvent that leaves no residue. Although expensive, "Freon" is recommended for such operations, because it leaves little or no residue and minimizes health and safety hazards associated with other solvents.

Oil and other contaminants which have infiltrated the surface of porous materials, such as cast iron, can be removed by heating. The temperature should be high enough to vaporize them and above the temperature the substrate will reach in spraying. Otherwise, bleeding or vaporization will occur during spraying.

If a workpiece is covered with scale from heat treating, or rust, it should be cleaned by abrasive blasting with sand or metal grit. The same treatment should be used for workpieces heated to remove infiltrated contaminants because they are likely to contain hard deposits after baking. The blasting operations should be conducted in equipment other than the unit assigned for use in preparing substrates for plasma spraying. That practice should be followed to prevent contamination of blasting media used for coating preparation. The parts should then be reblasted to a suitable surface roughness in the unit used for preparing substrates.

3.3 SURFACE ROUGHENING

After cleaning, one of several methods or combinations of methods should be used to produce a substrate to which a sprayed coating will adhere. The principal methods are (a) bond coating, (b) grit blasting, and (c) macro-roughening. To achieve an optimum surface for bonding, combinations of those methods are sometimes employed, such as bond coating and grit blasting, or machine roughening followed by grit blasting. Recent investigations indicate that shot peening before grit blasting offers advantages in some cases.

3.3.1 Bond Coating

Certain metals are able to adhere to a clean, smooth surface at moderate spray temperatures without the need for prior surface treatment. These bonding coat materials form strong coating-to-substrate bonds and are generally adherent over a wide range of conditions. For best results, however, it is desirable to grit blast the substrate before applying the bond, or intermediate coating, by plasma spraying. Bonding coats are extremely useful because a thin layer of one of these materials applied to a clean, smooth substrate can serve as a bond-enhancing subcoat for subsequently applied layers of other spray materials. Furthermore, a bonding coating applied to a grit-blasted surface will appreciably increase the bond strength of materials sprayed over it as compared to bond strengths of those materials applied to a grit-blasted surface without a bond coat.

Molybdenum is a commonly used bonding coat material; other materials with suitable properties, though not much used, are columbium and tantalum. Also very widely used is a commercially available nickel-aluminum composite which, when sprayed, undergoes an exothermic reaction to produce a nickel aluminide.

Certain general considerations should be observed in using a bond coat. Primarily, the decision to use a bond coat must take into account the conditions under which the coating will be used, in terms of temperatures or corrosive media. Molybdenum, for example, has poor oxidation resistance and would not be suitable for use in air at elevated temperatures. Nickel-aluminide is susceptible to corrosion in aqueous salt solutions.

The use of a self-bonding subcoat also limits the thickness of the coatings which may be applied, in that the surface it provides is generally not rough enough to absorb the shrinkage stresses produced by a thick coating. The bonding coat should be applied just thick enough to cover the substrate, usually no more than 0.002 to 0.004 inch thick. There is no advantage to spraying a heavy layer of bonding coat.

The selection of a bond coating is influenced by the composition of the substrate and the temperature the sprayed component will reach in subsequent processing or service. It is desirable that the elements in the bond coating be soluble in the matrix material of the substrate. The approximate maximum temperature limits for several commonly used bond coatings are:

Molybdenum	600 F	315 C
80Ni-20Al	1150 F	620 C
95.5Ni-4.5Al	1600 F	870 C
80Ni-20Cr	2300 F	1260 C

The NiCr Material is superior to the other bond coatings from the standpoint of resisting thermal shock.

3.3.2 Abrasive Blasting

Abrasive blasting is the most common method employed for roughening the substrate, after the workpiece is cleaned and/or shot peened, prior to plasma spraying. Abrasive blasting is accomplished by directing a stream containing abrasive particles against the surface of the substrate. The operation is conducted in order to remove surface contaminants such as oxide films or scale and to develop a surface finish suitable for bonding.

Abrasive particles used for blasting are classified as grit, sand, or shot. Grit consists of angular particles of steel, cast iron, malleable iron, or "sand", usually produced by crushing. The term sand is applied to many nonmetallic materials in addition to silica. Alumina, silicon carbide, pumice, and garnet are used for grit or sand blasting. Shot is made of the same metallic materials used for grit, but usually consists of roughly spherical or rounded particles. Some shot is made by cutting steel or aluminum wire and becomes more spherical during use.

Several types of equipment are used to propel abrasive particles in wet and dry blasting operations. Dry grit blasting is recommended for preparing surfaces for plasma spray coating. The operation is conducted with air blasting machines which force the abrasive particles through a nozzle with compressed air. Air-blast devices are more economical than mechanical equipment for processing small quantities of parts and for intermittent operations.

3.3.2.1 Grit Blasting

Manually controlled air blasting equipment is more convenient to use on small objects and is recommended for preparing surfaces to be thermal sprayed. Dry grit blasting operations are usually conducted in a cabinet that confines the abrasive dust, and houses the propelling mechanism and a suitable work-holding device or platform. The equipment is available in a variety of sizes. Figure 9 illustrates a common pressure type air-blasting device schematically. The grit is fed from a pressurized hopper to the stream of compressed, dry air and carried to the nozzle through a flexible hose. Figure 10 illustrates the principle employed in suction-type blasters to introduce the grit into the stream of pressurized air just ahead of the spray nozzle.

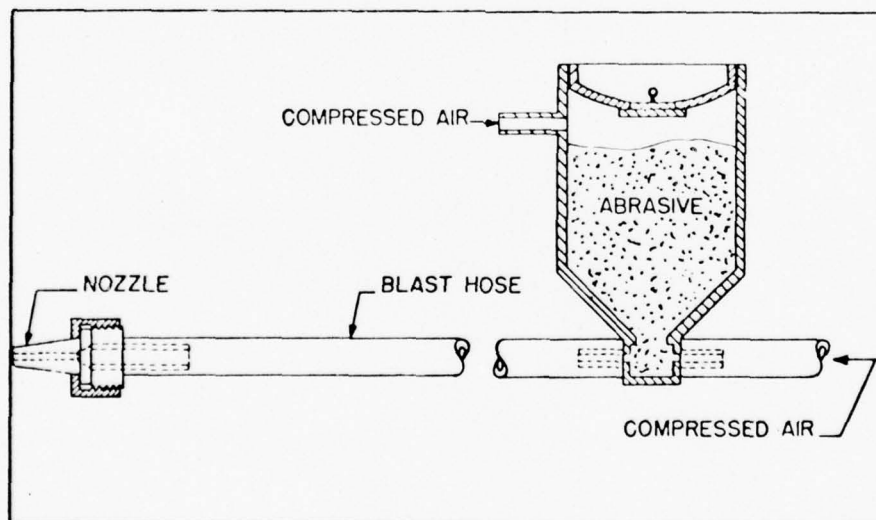


FIGURE 9. SCHEMATIC ILLUSTRATING PRINCIPLE OF A COMMON TYPE OF AIR BLASTING DEVICE

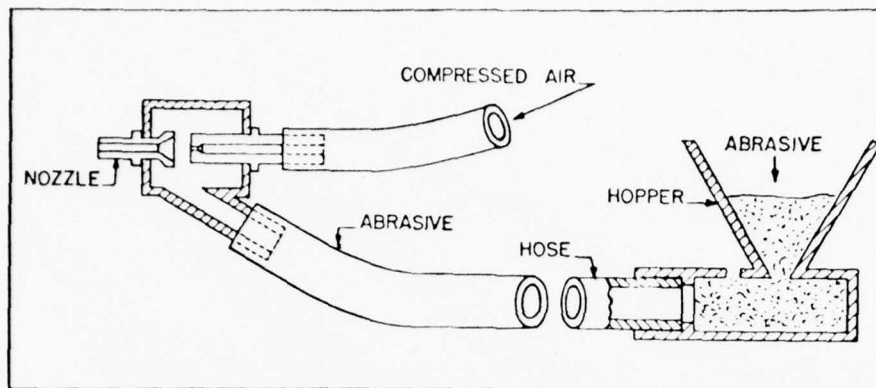


FIGURE 10. SKETCH ILLUSTRATING THE PRINCIPLE USED IN A SUCTION-TYPE AIR-BLASTING SYSTEM

Some commercially available equipment permits conducting air-blasting operations, particularly on large components, without a cabinet-type enclosure. During blasting, the device collects and removes the spent grit and dust by suction like a vacuum sweeper, as suggested by Figure 11. To do so, the blasting nozzle and its hose are surrounded by a flexible tube connected to a strong exhaust system. The suction hose is partially sealed to the surface of the workpiece, around the blasting nozzle, by a brush or flexible plastic cup. Although the method is comparatively slow and expensive, the approach is useful for grit blasting components too large to move or to fit into the cabinet available.

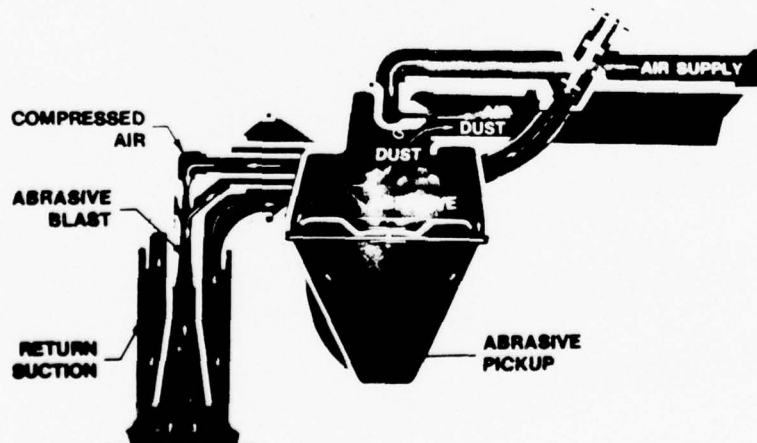


FIGURE 11. SCHEMATIC ILLUSTRATING PRINCIPLE OF AN AIR BLASTING SYSTEM THAT CAN BE EMPLOYED WITHOUT THE CABINET ORDINARILY USED TO CONFINED THE GRIT AND DUST (17)

Objects destined to be plasma sprayed should be grit blasted in units used only for that purpose. Blasting cabinets, and contaminated air lines, employed for abrasive blast cleaning of oily, rusty, or painted parts can produce surfaces unsuitable for plasma spraying. A blaster used for general purposes can be converted by thorough cleaning of the cabinet, replacing the abrasive, and putting in new air lines. The inside of the cabinet should be abrasive blasted and wiped with a solvent.

Grit blast nozzles erode quite rapidly even though they are made from hard cast iron, tungsten carbide, boron nitride, or alumina. A steel nozzle lined with boron carbide is usually a good choice, despite its higher cost, because it gives a much longer life. Because it wears more slowly, it saves air and provides more uniform spray patterns and particle velocities for a longer time than less-durable nozzles. A nozzle should be replaced when the outlet area has increased about 40 percent. In general, a nozzle used for air blasting metal grit will last about three times as long as one used with sand.

For reasons of safety, the carrier hose on blasting equipment should be inspected frequently and replaced when necessary. Hoses are most susceptible to wear at bends. The bending radii should be as generous as practical.

The effects of grit blasting depend on process parameters, of which size and type of abrasive, particle velocity, air pressure, blast angle, and blasting time are the most important.

The force of the impact of a single blasting particle depends on its change in momentum and with the mass and velocity of the particle. Smaller abrasive particles result in sprays giving more uniform coverage of the surface and smoother finishes. Heavier particles and higher velocities favor more rapid removal of material from the workpiece, as do harder materials. For otherwise similar conditions, higher air pressures produce slightly rougher surfaces.

Figure 12, from Golego and Panamarchuk, shows the effect on surface roughness of variations in spraying time and air pressure.⁽¹⁸⁾ The data were obtained in experiments on air blasting a titanium alloy containing 2.5 percent aluminum and 2 percent manganese with corundum grit particles ranging from 0.0013 to 0.0016 inch in size. Although the roughness values shown in the sketch are not standard AA values, and are presumed to be maximum peak to valley distances (which are ≈ 3.5 AA values), they illustrate several points. The graphs show that varying the air pressure for spraying times less than 30 seconds had no effect on roughness values. After full coverage is attained, continued blasting at the same pressure and grit size produces no change in microgeometry or roughness of a workpiece. The data also show that using higher air pressures for a particular grit size produces

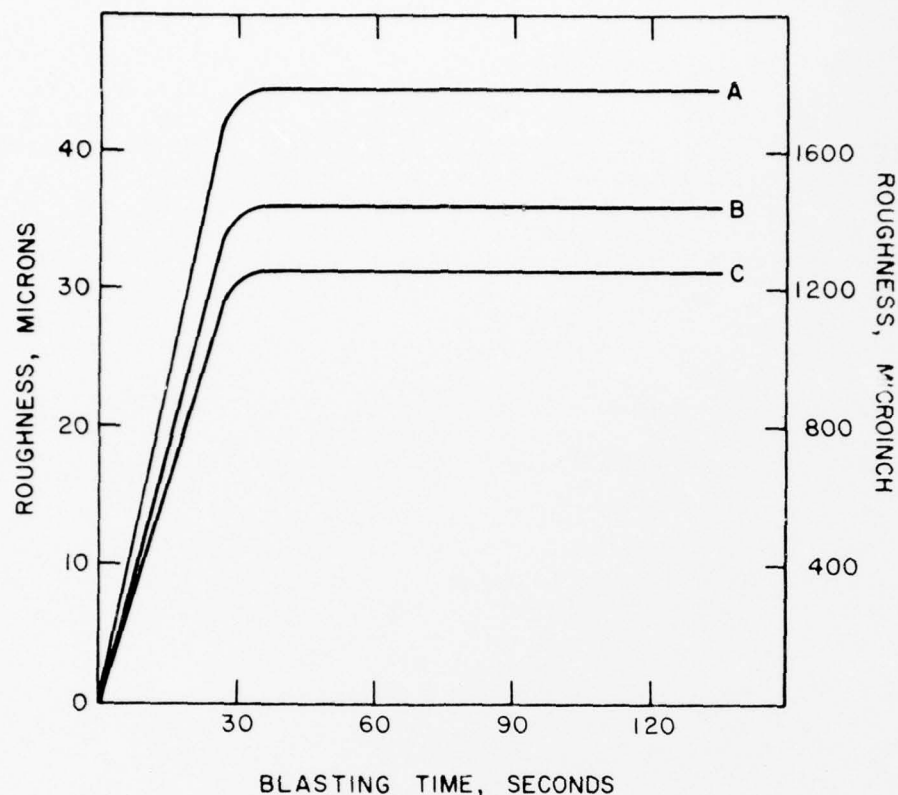


FIGURE 12. RELATIONSHIP OF SURFACE ROUGHNESS* OF A TITANIUM SUBSTRATE TO GRIT BLASTING TIME AND PRESSURE (A = 71.1 psi; B = 42.7 psi; C = 28.4 psi) (18)
* Presumably nonstandard method

slightly rougher surfaces. The authors also showed that the bond strength of a nickel-base coating, containing carbon, plasma sprayed to the titanium alloy substrate, varied with the roughness values identified in Figure 12. The relationship was as follows:

Roughness value, microns	400	80	1200	1600	2000
Bond strength, psi	1565	1850	2220	1960	1850

The data show that the best bond strength was associated with the roughness dimension comparable to three-fourths of the diameter (1200 microinch on the system used by the Russians or probably 350 AA) of the particles sprayed. The results seem to support the natural expectation that there is an optimum roughness value for good bonding and that the value should be related to the size of the particles sprayed.

Grisaffe also concluded that variations in surface roughness of grit blasted surfaces affected bond strengths.(19) His work, summarized in Table XVI, was conducted on alumina coatings, plasma sprayed (0.030 inch thick) on 304 stainless steel coatings. Although the strength values, determined with a nonstandard shearing device, appear low, they illustrate a principle. Within the ranges in roughness and powder-particle sizes investigated, the best bond strengths were obtained on substrates with the roughest surface tried. That surface was reported to have a roughness value of 280 microinch on the old rms system, which is approximately 420 microinch on the standard AA system. Other studies show somewhat similar effects, although the rate of improvement in bond strength with roughness apparently decreases above 375 microinch.

Smoother finishes must be used when the plasma coating is very thin, or for substrates that would deform if subjected to severe grit blasting. Since the roughness of the finish depends on the size of the grit, abrasives are furnished in different grades. The following suggestions will serve as a guide.

	Grit Size, mesh	Sieve openings, inch	Applications
Coarse	-10 +30	0.079/0.024	For coatings exceeding 0.010 inch and best adherence
Medium	-14 +40	0.056/0.017	For fair adherence and smoother finishes of coatings less than 0.010 inch thick
Fine	-30 +80	0.024/0.007	For smoothest finishes on coatings less than 0.010 inch thick to be used in as-sprayed condition.

Even new grit is made up of a range in sizes. Ordinarily, however, at least 80 percent of the particles will remain on a screen two sieve numbers higher than the smallest size mentioned above, e.g., 80 percent of the grit passing through the No. 14 screen would be expected to be retained by a No. 25 sieve.

Blocky, angular particles of alumina are the recommended abrasives for grit blasting substrates to be plasma sprayed. Silicon carbide grit is also suitable, but is more expensive. A blasting pressure on the order of 80 psi is normally appropriate for hard substrates. Lighter pressures should be used for softer workpieces such as aluminum, copper, their alloys, and bronzes. That practice will minimize the likelihood of embedding particles of the grit in the substrate. Dust adhering to the workpiece, from the grit blasting operation, should be removed by air blasting or brushing before starting the plasma spraying operation.

TABLE XVI. BOND STRENGTH DATA FOR ALUMINA COATINGS
PLASMA SPRAYED^(a) ONTO STEEL SUBSTRATES
WITH VARIOUS ROUGHNESSES⁽¹⁹⁾

Powder ^(b)	Purity	Powder Size, microinch		Surface Roughness, microinch		Median Bond Strength, ^(e) psi
		Range	Median	rms ^(c)	AA ^(d)	
A	97.5	156/1840	750	280	420	545
B	98.0	148/1580	350	115	170	270
				225	340	390
				280	420	440
C	99.49	292/3280	456	280	420	510
D	99.49	136/1520	635	115	170	380
				225	340	540
				280	420	600

(a) Arc current, 400 amperes; arc voltage, 70 volts; powder feed rate, 2 lb/hr; torch to substrate distance, 6 in.; arc gas, 100 cu ft dry nitrogen and 15 cu ft hydrogen/hr; nitrogen carrier gas flow, 10 cu ft/hr.

(b) All powders determined to be 100 percent alpha alumina by X-ray diffraction.

(c) Roughness values reported in nonstandard rms values.

(d) Roughness values converted to standard values by an approximation.

(e) Determined by a shearing test.

Typically, nozzle-to-substrate distances of 2 to 6 inches are used for grit blasting. Variations within that range seem to have little effect on roughness, but may affect coverage. Impingement angles near 90° give best results.

There is no dependable formula for predicting the minimum time needed to produce a grit-blasted surface suitable for plasma spraying. A suitable blasting time should be established by experiment with the equipment to be used in production and with specimens comparable to the substrate of interest. The time can be judged by visual inspection for uniform coverage and surface texture.

3.3.2.2 Shot Peening

Peening is a cold working process that deforms the material near the surface of the workpiece and creates compressive stresses in that region. Shot peening, like grit blasting, is accomplished by a high-velocity stream of metal particles suspended in compressed air directed at the metal substrate. The impact of individual particles of shot creates rounded depressions in the surface and deformation to a depth of 0.005 to 0.010 inch below the surface of the substrate. Compressive stresses are set up in the deformed material because it has a larger surface area and is attached to the unstrained material deeper in the substrate. Controlled, severe peening can produce compressive stresses at the surface ranging up to half the yield strength of hard metals. Such residual stresses are desirable when the surface layers will be subject to tensile stress in subsequent operations or service. The beneficial effects of compressive residual stresses in bending fatigue are well known. It has been established rather recently that shot peening is also a desirable and beneficial method of preparing surfaces for plasma-arc spraying.

Data obtained by Bethke of NADC-Warminster on the effects of surface preparation methods on fatigue properties of high strength steel, with and without plasma-sprayed coatings, are presented in Table XVII.⁽²⁰⁾ The data were obtained on rotating-beam fatigue specimens (0.250 X 3.42 inch gage length) of 4340 steel, heat treated to 280,000 psi. Precautions were taken during plasma spraying to avoid heating the substrates above 275 F. Fatigue tests were made at two stress levels in order to determine the effects of processing variables. As is normally the case, shot peening improved the fatigue life of the steel appreciably in both short and long cycle fatigue. Grit blasting caused much less improvement than peening. More importantly, the data also show that specimens coated after grit blasting had poor fatigue properties, lower than the base metal. On the other hand, specimens that had been shot peened, then grit blasted and coated by plasma spraying had very good fatigue properties. The data indicate that peening should be seriously considered as a method for preparing surfaces of components when the highest-quality plasma-sprayed coatings are required on components subject to fatigue.

Shot peening is normally performed by dry blasting with cast steel shot having a hardness of Rockwell C 40 to 50. Glass beads are often used for peening materials that would be contaminated by embedded iron such as aluminum, titanium, or stainless steel. Workpiece areas that should not be peened should be protected from the blast by masks.

TABLE XVII. EFFECTS OF SURFACE PREPARATION TREATMENTS AND PLASMA SPRAYED COATINGS ON FATIGUE LIFE OF 4340 STEEL SPECIMENS IN ROTATING BEAM TESTS (20)

Specimen Condition	Stress Level, 180 ksi		Stress Level, 110 ksi	
	Life, cycles (a)	Improvement, percent	Life, cycles (b)	Improvement, percent
Base Material (c)	11,500	Control	448,000	Control
Peened (d)	18,000	57	4,509,000	906
Grit Blasted (d)	13,500	22	1,135,000	153
Peened and Grit Blasted (d)	17,500	52	3,960,000	780
Blasted and Coated (e)	11,500	0	251,000	-44
Peened, Blasted, Coated (e)	20,500	78	3,476,000	+675
Peened, Blasted, Coated (f)	-	-	2,168,000	+383

(a) Average for 2 specimens.

(b) Average for 3 specimens, 5 for control.

(c) 4340 steel heat treated to a tensile strength of 270 ksi, 0.250-inch fatigue specimens polished on 600-grit paper.

(d) Peening with 230 steel shot to an Almen intensity of C10-12, blasting with 54 mesh alumina grit at 60 psi pressure.

(e) Coated with nickel aluminide (Metco 404) to a thickness of approximately 0.026 \pm 0.0004 inch.

(f) Coated with a mixture of nickel aluminide and tungsten carbide (Metco 439) to a thickness of approximately 0.0018 inch.

Cast metal shot for peening operations is available in different sizes; those identified in Table XVIII can be used for most application. The shot size number denotes the diameter, in ten thousandths of an inch, of the major fraction of the particles.

TABLE XVIII. TYPICAL SPECIFICATIONS FOR CAST IRON AND STEEL SHOT FOR SHOT PEENING (21)

Shot Size Number	Cumulative Sieve Fraction, percent	Screen Opening, in.	Screen Number
S 280	0	0.0469	16
	5 max. on	0.0394	18
	85 min. on	0.0280	25
	96 min. on	0.0232	30
S 230	0	0.0394	18
	10 max. on	0.0331	20
	85 min. on	0.0232	30
	97 min. on	0.0197	35
S 170	0	0.0331	20
	10 max. on	0.0280	25
	85 min. on	0.0165	40
	97 min. on	0.0138	45
S 110	0	0.0232	30
	10 max. on	0.0197	35
	80 min. on	0.0117	50
	90 min. on	0.0070	80
S 70	0	0.0165	40
	10 max. on	0.0138	45
	80 min. on	0.0070	80
	90 min. on	0.0049	120

The effects of shot peening on deformation and residual stresses, the characteristics conferring benefits, depend on coverage and peening intensity. The depth of the deformed layer increases with peening intensity. The lowest peening intensity providing the desired effect on the workpiece is most efficient and least expensive. Methods of evaluating peening intensity for conditions of interest, by measuring effects produced on a standard specimen, are described in the section on quality control. Intensity tests should be made at appropriate intervals to check whether conditions for producing selected peening intensities with available equipment have changed with time.

Surface coverage, sometimes called saturation, is a measure of how completely an area has been hit by the shot particles. Unless coverage is adequately complete, the full benefits of peening will not be obtained. Various methods have been used to judge the extent of coverage by peening. Visual examination is widely used and adequate for judging the operation as a method for preparing a surface for plasma spraying. Fortunately, it has been established that the coverage for short and long spraying times follows the following relationships(21):

$$C_n = 1 - (1-C_1)^n$$

where C_n = % coverage, expressed as a decimal, after peening for n cycles

C_1 = % coverage, expressed as a decimal, after peening for 1 cycle.

For instance, if peening for a 1-minute cycle produced 25 percent coverage ($C_1 = 0.25$), peening for 6 or 12 minutes is expected to give coverages of 82 or 96.8 percent coverages, respectively. Arbitrarily, 98 percent coverage is considered complete saturation; coverage values beyond 98 percent are expressed as multiples of that time. For instance, a value of 1.5 means the specimen was peened 1.5 times as long as the period required for full coverage.

Changes in air-blasting parameters that affect particle velocities also affect peening intensities. The experimental data in Table XIX show that increasing the spacing between the nozzle from 4 to 8 inches decreased the peening intensity appreciably. For the conditions investigated with the 1/2-inch nozzle, increasing the aspirator size considerably increased the peening intensity. The aspirator setting was less important for the other two nozzle sizes.

The angle between the direction of the stream of particles and the surface of the substrate is known as the impingement angle. The maximum peening intensity is attained with an impingement angle of 90°. Decreasing the impingement angle lowers the peening intensity by an amount proportional to the sine of the angle. That is, for otherwise similar conditions, the effect of impingement angles is:

Impingement angle	90°	60°	45°	30°
Relative peening intensity	1.0	0.87	0.71	0.50

TABLE XIX. EFFECT OF NOZZLE SIZE, ASPIRATOR SIZE,
AND NOZZLE-WORK SPACING ON INTENSITY
OF SHOT PEENING(a) (21)

Nozzle Diameter, inch	Aspirator Size, inch	Peening Intensity, (b) for Nozzle-Work Distance Indicated		
		4 inch	6 inch	8 inch
3/8	5/32	5	5	5.5
	7/32	5	5	4
1/2	5/32	3.5	3	3
	7/32	7	7	5
5/8	5/32	7.5	5.5	7
	9/32	7.5	7	6

(a) Tests made with suction-type shot peening test cabinet, S 70 steel shot, on 410 stainless steel blades; blast pressure 92 psi, flow of shot to nozzle, 7-1/4 lb/min.

(b) Larger numbers indicate greater peening intensity. Values indicate arc heights, in thousandth of an inch, for A-type Almen test strips.

3.3.3 Macro-Roughening

Macro-roughening is a term used to describe methods for producing large changes in surface topography to increase surface area and reduce stresses induced by coating shrinkage. It involves cutting grooves or deep striations into the substrate and is used in instances where thick coatings over 0.030 in. are required.

This method is most often used in preparing cylindrical sections for coating. The procedure usually followed is to mount the cylindrical section in a lathe and cut deep, closely spaced screw threads into the surface. After threading, the part is grit blasted and/or coated with a bonding layer of molybdenum or nickel-aluminide.

On flat surfaces, the surface may be rough milled or grooves or slots may be cut into the edges. This would again be followed by grit blasting and/or bond coating.

SECTION IV

4.

MASKING TECHNIQUES AND MATERIALS

In many applications, only specific areas of a workpiece may need to be coated. In such cases, the areas adjacent to the surface to be coated must be protected, both from unnecessary surface roughening and from adherence of the sprayed material. The technique of protecting the areas adjacent to the surface to be grit blasted and/or plasma sprayed is masking. Separate types of masking may be required for grit blasting and for spraying, though in many instances the same mask may be used for both operations. Some masking materials used with success are listed in Table XX.

4.1

MASKING FOR GRIT BLASTING

For most grit-blast masking applications, ordinary one-inch-wide paper masking tape, applied several layers thick, affords satisfactory protection to adjacent surfaces. It has the advantages of being inexpensive and easy to cut or shape as needed. However, since it is easily cut, it may be damaged in a heavy or prolonged blasting operation, resulting in grit penetration or edge erosion, and consequent damage to the substrate. Also, this type of tape should be removed prior to spraying to avoid coating contamination or excessive build-up at the coating edge.

Adhesive-backed cloth, plastic, or rubber tapes or Teflon are other suitable masking materials. They are easy to apply, provide protection, and are less likely to be damaged by abrasive blasting. All of these materials should be removed before plasma spraying.

Of the various types of tape available for masking, the most suitable is the heat-resistant, metallic-coated cloth tape, such as is used in many furnace or heating applications. This material is also easy to apply to the areas to be protected, it is not severely damaged in grit blasting, and can be left in place to serve as a mask during coating.

For high-production grit blasting, taping is too slow a process for masking. In this case metal or rubber shields may be used, provided the geometry of the workpiece is not too complex. Of the two materials, rubber is the better choice as grit blasting mask because it is not cut by the grit blast abrasive, whereas a metal mask will ultimately erode. However a metal grit blasting mask may be used as a thermal spray mask as well, while rubber cannot. The use of metal or rubber shield is not economical except in high production applications.

TABLE XX. DESCRIPTION OF MASKING MATERIALS

Description	Product Identification	Form	Method of Application	Masking For		Method of Removal
				Grit Blasting	Plasma Spraying	
Teflon-fiber-glass resin backed tape	Borden's Mystik #7010	Tape	Manual	X	X	Peel
Fiber glass tape	3M Scotch #69	Tape	Manual		X	Peel
Vinyl tape	3M Scotch #33	Tape	Manual	X		Peel
Chlorinated hydrocarbon (7% toluene solvent)	Torco 522-66	Liquid	Dip or brush	X	X	Soften in solvent (peel)
Proprietary	Nicrobraz Green Stop-Off	Liquid	Brush or spray (aerosol can)		X	Mechanical (brushing)
Proprietary	Metco Antibond	Liquid	Brush		X	Water wash or brush
Water soluble ink	Printers' ink	Liquid	Dip or brush		X	Water-wash-brush
Commercial grade Teflon	Teflon	Rod-sheet-plate	Cut as templates and place manually	--	X	--

The use of tape or of metal or rubber shields is generally limited to flat or cylindrical surfaces. In instances where holes, slots, keyways, or other types of recesses must be protected, inserts of carbon or metal should be used. These are left in place during grit blasting and spraying, and usually are not removed until completion of surface finishing.

4.2 Masking for Plasma Spraying

Basically, the same methods are used to mask for spraying as are used in masking for grit blasting; the difference is in the choice of materials. As pointed out earlier, most grit blast masking tapes must be removed prior to spraying. They may burn or char, or the adhesive backing may run. Combustion products or soot may contaminate the coating. The exception is glass-fiber tape, made for high-temperature use. It can withstand the spray temperatures encountered in most coating operations and is a convenient and inexpensive material.

Where geometric considerations permit, smooth metal masks may be used. Again, this method is best suited to a high-production operation. Provision must be made, when using metal masks, to prevent coating build-up on them. This may require that they be water cooled, coated with a suitable "stop-off" material, or equipped with some mechanical means of removing deposited material.

Another masking technique suitable for thermal spray applications is the use of liquid masking compound. These compounds, when applied over blasted areas adjacent to the desired deposit area, act as stop-offs, to prevent the adherence of the sprayed powders to the base metal in these areas. They may be applied by painting or dipping following grit blasting, making them rather inexpensive to use.

Threaded holes, slots, and keyways can be protected from thermal spraying by metal, Teflon, or carbon inserts, left in place until after completion of finishing operations. In such areas, particularly around threaded holes, it may be desired that the sprayed coating not end sharply, but rather taper to the edge of the hole or some point near the edge. In such cases a technique referred to as shadow masking is used. In shadow masking, the area to be sprayed is partially shielded, by raising the mask a small distance off the surface, thus permitting some overspray to extend under the mask to produce a feathering at the coating edge rather than a distinct boundary.

SECTION V

5.

PLASMA-SPRAY DEPOSITION VARIABLES

In theory at least, a thorough knowledge of the effects of parametric variables of the plasma-spray process would permit tailoring the characteristics of a coating to a specific application. For example, coatings with an appropriate amount of porosity may be desirable for retaining lubricants and minimizing wear under some service conditions. For other applications high-density coatings are preferable. Unfortunately, the state of the art does not yet permit predicting the precise process parameters necessary for producing coatings with the optimum characteristics for specific applications. The problems result from the fact that many variables affect the results of the plasma-spray process and their interactions have not been established quantitatively. For instance, several types of equipment are marketed and used for the deposition process and they differ significantly in design and operating characteristics. The control settings, especially the selection of voltages and currents, appropriate for producing satisfactory coatings vary with the plasma-arc units as well as with other parameters chosen by the operator. Under the circumstances, the most practical approach for choosing the operating conditions best suited to a particular application is to use the recommendations of the gun and powder suppliers as a guide. For a combination of substrate and coating new to the operator, the preliminary trial is best made on a specimen rather than an actual component. If any serious deficiencies in characteristics of the coating are noted on the trial specimen, it is desirable to conduct additional experiments with slightly different process parameters. In order to be helpful, some information on the effects of changing parametric variables on process and product characteristics are given in this section. When the conditions considered most suitable have been selected, they should be closely reproduced in plasma spraying the components to be used in service. To do so, the use of automated or semiautomated control of gun movements and other process variables is strongly recommended. The control of process variables is too critical and difficult to be achieved by manual spraying.

In general, the information in this section is concerned with producing coatings of the high quality needed for severe service in aircraft and Naval Ordnance. The selection of plasma-spraying conditions depends more on the properties of the material to be deposited than on the properties of the substrate. Nevertheless, it should be noted that the substrates of greatest interest were identified by Naval Ordnance personnel as:

Carbon steel	Aluminum bronze
Low-alloy steel	Valve bronzes
Stainless steel	K-Monel
Aluminum alloys	Brass

The section on data sheets, in this report, lists some specific conditions found suitable for depositing good coatings with one particular type of plasma-spray gun. Those parameters should be adjusted when other types of equipment are employed.

Figure 13 shows the modules constituting a plasma-arc spraying system and suggests the variables that must be controlled. The power level, the pressure and flow of the arc gases, and the rate of flow of powder and carrier gas are controlled at the console of the system. The gun orientation and gun-to-work distance are usually preset, and the movement of the workpiece is ordinarily controlled by using automated or semiautomated tooling. Substrate temperatures should be controlled by preheating and by limiting the temperature changes during processing.

5.1 ARC GASES

The selection of the arc gas depends on the type of material to be sprayed and to some extent on the type of plasma-arc equipment to be used. All equipment and most spray-material suppliers give recommendations as to the types of arc gases to be used with particular types of powders.

Generally a nonoxidizing gas is required to form the plasma and the inert gases helium and argon are often used. Nitrogen is also a common arc gas, except for spraying materials that would be embrittled by contamination with nitrogen. The principal advantage is that diatomic gases such as nitrogen and hydrogen have higher heat contents, at the same high temperature, than helium or argon. That point is illustrated in Figure 14, attributed to Dunham by Fisher.⁽²²⁾ Higher heat contents are particularly desirable for spraying refractory materials such as oxides, if care is taken to prevent overheating the substrates. On the other hand, if the heat content is unusually high, nozzle erosion may be severe. Therefore, it is common practice to use argon as the primary arc gas and use some hydrogen or nitrogen as the secondary gas to form the plasma. Frequently mixtures of 5 to 25 percent hydrogen are used for the arc gas, with the primary gas being argon or nitrogen. Argon is preferable to avoid trouble from contamination or excessive porosity.

The composition of the arc gas influences the arc voltage and the power level. A higher current is needed for maintaining a particular power level for pure-argon arc gas than for mixtures of argon with hydrogen or nitrogen.

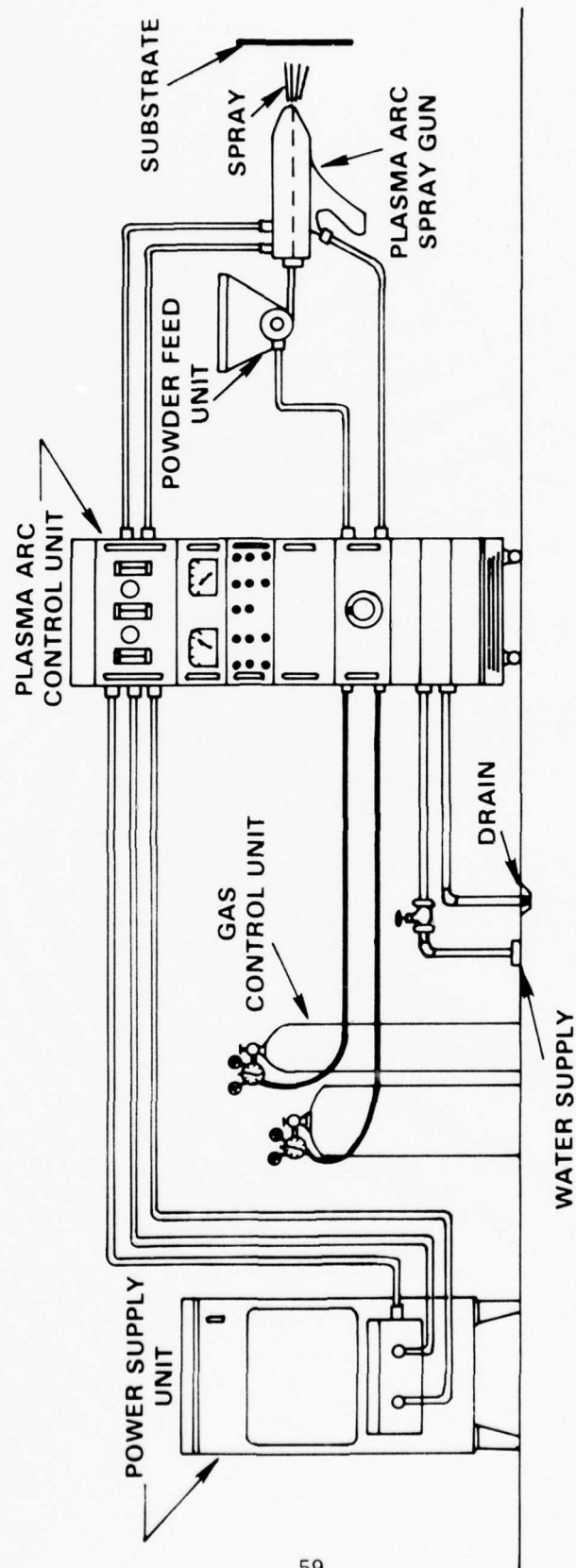


FIGURE 13. SCHEMATIC OF PLASMA-SPRAY SYSTEM

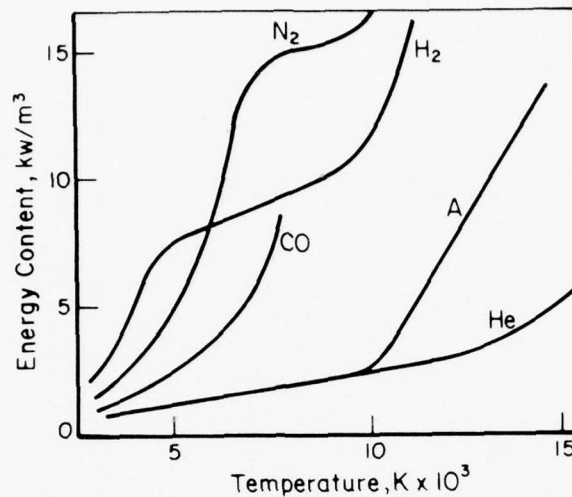


FIGURE 14. ENERGY CONTENT OF GASES AT DIFFERENT TEMPERATURES, ADAPTED FROM DATA BY DUNHAM BY FISHER⁽²²⁾
(Temperature $^{\circ}\text{K} = ^{\circ}\text{C} + 273$)

The initial and partial control for the flow of arc gas is provided by a two-stage, high-pressure regulator at the gas supply, as suggested in Figure 13. The finer and more critical control is established by setting the flow meters on the plasma system console. Those flow meters are calibrated to operate from specific input pressures specified by the system manufacturers. Therefore, it is essential that the two-stage regulators be set properly. It is, of course, equally important to set properly the flow meters to deliver the flow specified by the parameter or process instructions for spraying a given material. It is of particular importance to obtain the proper flow of secondary arc gas, as this setting controls the voltage value.

Excessive flow rates of the arc gases and, in particular, the primary arc gas will remove useful heat from the plasma stream and at the same time increase spray particle velocity which, in turn, reduces the dwell time of the spray particles in the plasma stream. As a consequence, much of the spray material will not attain the semimolten or plastic state necessary to achieve the deformation which permits the powder particles to adhere to the base metal and to cohere to one another. The resultant coating will lack proper bond strength, coating strength, density, and hardness. Additionally, there will be a marked decrease in deposit efficiency.

Conversely, low arc gas flow rates result in improper voltage values and greatly reduced particle velocities. In extreme cases, insufficient arc gas flow can result in overheating of the spray material particles to the point that the material will be vaporized or the spray material will melt prematurely and plug the powder injection part at the point of injection into the plasma stream. In addition, a very low arc gas flow can result in the collection of molten powder particles in the spray nozzle or powder injection port and subsequently depositing this material into the coating as a large globule. Upon grinding or masking the coating, this globule will result in a rather large coating void. If the spray operator observes a large globule of spray material being deposited onto the coating, he may be able to remove it, providing the spray operation can be stopped before a great deal of additional material is deposited over the globule.

Finally, under the proper circumstance, a very low arc gas flow will result in damage to the spray device in the form of extremely heavy erosion of the spray nozzle or destruction of the spray gun housing as a result of internal arcing.

The qualitative effects of varying the flow rate on deposition efficiency and on coating density are shown in Figures 15 and 16. The sketches illustrate the effects expected when other spraying variables are held constant. It should be noted that there is an optimum rate for gas flow and that the optimum rate varies with other spraying variables.

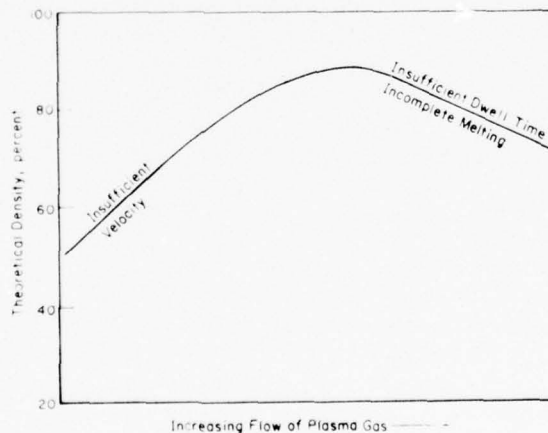


FIGURE 15. THE EFFECT OF ARC GAS FLOW ON COATING DENSITY FOR OTHERWISE CONSTANT CONDITIONS (23)

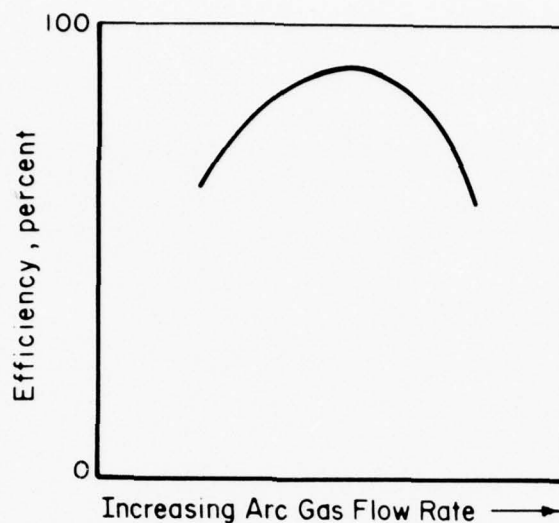


FIGURE 16. EFFECT OF ARC GAS FLOW RATE ON SPRAYING EFFICIENCY, ADAPTED FROM DATA BY ATKINS BY FISHER⁽²²⁾

After the arc gas and cooling water are turned on and the flow rates adjusted to the desired levels, the arc can be initiated. Most control systems are interlocked so the equipment will not operate without a flow of coolant. The voltage of the d-c arc depends on the gun design and the plasma forming gas.

5.2 ARC POWER LEVEL

The maximum power rating of a plasma gun is limited by its design, principally by the efficiency of the system for cooling the electrode. The power level of the arc, expressed in kW, is the product of the voltage and the current in amperes. The power level controls the temperature and influences the heat content of the arc. Higher power levels are useful in spraying materials with higher melting points.

The control of voltage is a function of the type and flow rate of the arc gases, primarily of the secondary arc gas. The voltage can be raised or lowered by increasing or decreasing the flow rate of the secondary gas which is usually either hydrogen or helium. Some authorities do not recommend increasing the hydrogen flow rate to increase the voltage.

Failure to attain the voltage value specified by the process instruction sheet within the arc gas flow limits established by the process instruction sheet usually indicates that the arc

length is too long and that the front and rear electrodes in the plasma gun should be replaced because of erosion. Since the arc length is proportional to voltage, rotating the nozzle will sometimes restore the voltage to the desired level because the arc will travel to an uneroded spot.

If the voltage is within the recommended range but the power level is outside the desired range, the current should be adjusted. Figure 17 indicates that there is an optimum power level for otherwise constant spraying conditions. The optimum power is particularly sensitive to powder composition and particle size because those characteristics set the heating requirements. Even though the desired power level is attained on starting the plasma-spray system, the console indicators should be monitored. This prevents undetected changes caused by fluctuations in line voltage, changes in flow of the secondary arc gas, or erosion of the electrodes. Settings should be changed when necessary.

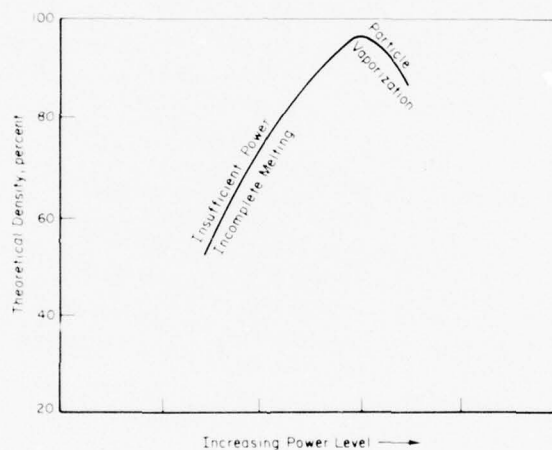


FIGURE 17. EFFECT OF ARC-POWER LEVEL ON DENSITY OF PLASMA-SPRAYED COATINGS FOR OTHERWISE CONSTANT CONDITIONS (23)

Using power levels below the optimum results in inadequate heating of sprayed particles, lower bond strengths, coating strengths, hardnesses, and deposition efficiencies.

Exceeding the optimum arc-power levels raises costs by wasting power and accelerating deterioration of gun components. Excessive power can vaporize some of the spray material and cause changes in composition. Condensation of the vapor on the substrate, or between layers of laminated coatings, results in poor bonding.

The size of the particles used for plasma-arc spraying is important. The size affects the optimum power level setting and gas flow rate because larger particles require more heat and longer dwell times for melting. Particle size also influences coating quality because it affects the kinetic energy of the moving particles at the time they hit the substrate. Excessively fine particles decelerate rapidly during spraying and produce coatings with lower densities and poorer deposition efficiencies.

Experience indicates that the optimum particle size is ordinarily in the range of 140 to 325 mesh (0.0041-0.0017 inch). Some investigators report that larger particles in that size range are preferable because they are better able to hold heat and to deform around surface irregularities to give better bond strengths. Any supply of powder consists of particles of different sizes. Whether the particle-size distribution should be wide or narrow within the limits mentioned is not known with certainty. Contradictory results have been reported by different investigators. Some favor a narrow range to promote chemical uniformity. Others prefer a more uniform distribution of different sizes and recommend blending of powders to achieve it, in an effort to obtain better densities and bond strengths. Table XXI lists the particle size ranges of some powders used successfully for plasma-arc spraying.

In establishing suitable plasma-arc spraying parameters for a particular material, a consistent feed rate is of the utmost importance. This factor must be controlled to determine the level of power required to provide adequate particle melting. Should the powder feed rate exceed the value used to establish the power setting, the particles will not be heated adequately, resulting in a weak, porous structure and a lower deposition efficiency. Conversely, lower material feed rates will result in overheating and vaporization of the spray material.

All commercial feed devices are capable of rather precise control (within 2 percent) of the amount of spray material fed through the unit in a given period. The feed rate is generally expressed in pounds per hour (lb/hr). Powder feeders use wheels or screws to deliver a fixed volume of material from the hopper to the stream of carrier gas. Consequently, the weight of powder fed at a particular setting increases with the bulk density of the material. Variations in composition, shape (spherical, flake, or angular particles), and size of powders affect bulk density and feeding rates based on weight.

It is extremely important to blend the spray material properly to eliminate segregation or stratification by particle size, which can occur during shipment or storage. The finer particles will generally settle to the bottom of the container, forming layers characterized by different particle sizes. Proper

TABLE XXI. PLASMA-SPRAY POWDER SPECIFICATIONS

Material	Description	Supplier	Particle Size(a)
Aluminum oxide - titanium oxide composite, Al ₂ O ₃ -TiO ₂ (Metco 130)	87% Al ₂ O ₃ 13% TiO ₂	Metco, Incorporated Westbury, Long Island, NY	-270 mesh + 15 μ (-53 + 15 μ)
Chromium oxide, Cr ₂ O ₃ (Metco 106 NS)	99% Cr ₂ O ₃	Metco, Incorporated	-140 mesh + 10 μ (-106 + 10 μ)
(AVCO PP-39)	98% Cr ₂ O ₃	Bay State Abrasives, Westboro, MA	-325 mesh + 10 μ (-44 + 10 μ)
(Plasmadyne 309F)	97% Cr ₂ O ₃	Plasmadyne Santa Ana, CA	-325 mesh (-44 μ)
Molybdenum, Mo (Metco 63 NS)	99% Mo	Metco, Incorporated	-200 mesh + 30 μ (-75 + 30 μ)
Nickel-chrome alloy, Nichrome (Metco 43C)	80% Ni 20% Cr	Metco, Incorporated	-140 + 325 mesh (-106 + 45 μ)
Nickel-aluminum composite, Nickel aluminide (Metco 450)	4.5% Al 95.5% Ni	Metco, Incorporated	-170 + 325 mesh (-90 + 45 μ)

blending eliminates this condition, which otherwise produces a gradual change in bulk density from top to bottom of the powder hopper, and thus a change in actual feed rate.

Although one can be reasonably certain of repeatability of the material delivery rate at the point the material leaves the feeder, other factors can seriously affect both the quantity and consistency of the feed rate at the point of injection into the plasma stream:

- (1) Length and diameter of the powder feed line, and
- (2) Spray material form and size.

Generally speaking, if the standard spray material feed lines supplied by the spray device manufacturer are utilized, the powder feed line will probably not cause feeding problems, providing there are no sharp bends in the line as a result of the manner in which the unit is set up.

Frequently, it has been noted that spray equipment users will improvise material feed lines. Changes in either the length or interior diameter of the powder feed line will result in either or both a lowering of the feed rate or, worse, a pulsation in the feed rate. Should the spray material be injected into the plasma stream in bursts, relatively improperly heated particles will be deposited into the coating, resulting in a loosely bonded porous structure with areas of varying hardness.

Feed rates are affected by moisture contents of the powder and by erosion of the injection ports or other components. Powder-feeding rates are influenced to a minor extent by the flow rate of the carrier gas. More importantly, if the gas flow rate is too high, transit times are shorter and particles will not be heated thoroughly by the plasma stream.

5.4 GUN-TO-WORK DISTANCE

The gun-to-work or spray nozzle-to-work distance primarily affects the following plasma-arc spraying parameters:

- (1) Spray particle velocity at the point of impingement on the substrate or coating.
- (2) Spray particle temperature at the point of impingement on the coating or substrate.
- (3) The temperature of both substrate and coating.

The characteristics of the coatings are very sensitive to the gun-workpiece distance. Depending on the equipment, power level, material combinations, and other conditions, there is an optimum spacing that must be determined by experiment. Most materials are sprayed at gun-workpiece distances ranging from 2 to 5 inches; the

spacing considered suitable should be controlled within 0.2 inch for critical applications. Such precision is difficult or impossible with hand-held guns.

It can readily be appreciated that the spray particle velocity will diminish as the particles travel from the spray nozzle to the workpiece. They also start to cool as they leave the stream of plasma. Therefore, excessive spray nozzle-to-work distances result in less particle deformation and, consequently, in loosely bonded, porous and weak coating structures, and a substantial loss in deposition efficiency.

Too short a spray nozzle-to-work distance is probably more destructive to coating properties than an excessive distance. Certainly it can be stated that the results are more pronounced and instantly obvious. Severe oxidation of the substrate and coating will occur. This, in turn, weakens or, depending on the degree of oxidation, completely destroys the substrate-to-coating bond. Additionally, the substrate would naturally expand as a function of coefficient of thermal expansion and, depending on the match between the coefficients of thermal expansion between substrate and coating, spalling could occur.

It should be noted, however, that if adequate coating and substrate cooling can be maintained, extremely dense coatings will result from using the minimum practical spray nozzle-work distances. One of the more effective methods of cooling for very close gun distances is the use of liquid CO₂ sprayed onto the part both immediately preceding and following the point of spray impingement.

It is strongly recommended that the spray nozzle-work distance be controlled by mechanical fixturing. Only by the use of automated spray tooling can consistent coating quality be assured.

Table XXII illustrates the effect of different gun-to-work distances on the porosity of plasma-sprayed coatings when other deposition variables are held constant. The data were obtained by Nimvitskaya and Fishman in experimental coating of carbon steel with aluminum oxide, using Russian equipment.⁽²⁴⁾ The porosity of the alumina coating was measured by filling the pores with a standardized procedure and weighing. That technique probably determines only the volume of interconnected voids open to the surface.

5.5 SPRAY ANGLE

The spray angle designates the attitude of the spray nozzle in relationship to the substrate. As indicated in Figure 18, the plasma stream is ordinarily directed at the substrate at a spray angle of 90°. Spraying perpendicular to the substrate gives better deposition efficiencies. In theory, that spray angle should also result in better bond and coating strengths, but those points have not been investigated extensively. Unless it is substantiated

TABLE XXII. EFFECTS OF ARC GAS AND OF GUN-WORKPIECE DISTANCE ON POROSITY OF PLASMA-SPRAYED ALUMINA COATING (24)

Arc Gas and Pressures (a)	Gun-Work Distance, in.	Coating Porosity, percent
Argon, 28.5 psi	4	7.75
+ Nitrogen, 25.7 psi	6	21.6
	8	44.3
Nitrogen, 28.5 psi	6	27.6
Nitrogen, 28.5 psi	4	4.35
+ Helium, 14.3 psi	8	9.8
Nitrogen, 28.5 psi	4	12.1
+ Helium, 28.5 psi	6	9.7
	8	25.8

(a) Pressure in powder feeder, 1.46 psi.

by testing for a given application or required for a particular workpiece configuration, the spray nozzle should not be set at greater than a 15-degree angle from the perpendicular to the workpiece.

For some applications where maximum coating and density is not required, satisfactory results have been obtained with a spray angle as large as 45 degrees. Decisions on spray angles should be based on tests with appropriate specimens and metallographic examination.

The prime concern in spraying at too great an angle is that the spray particles will tend to skid rather than flatten upon impacting.

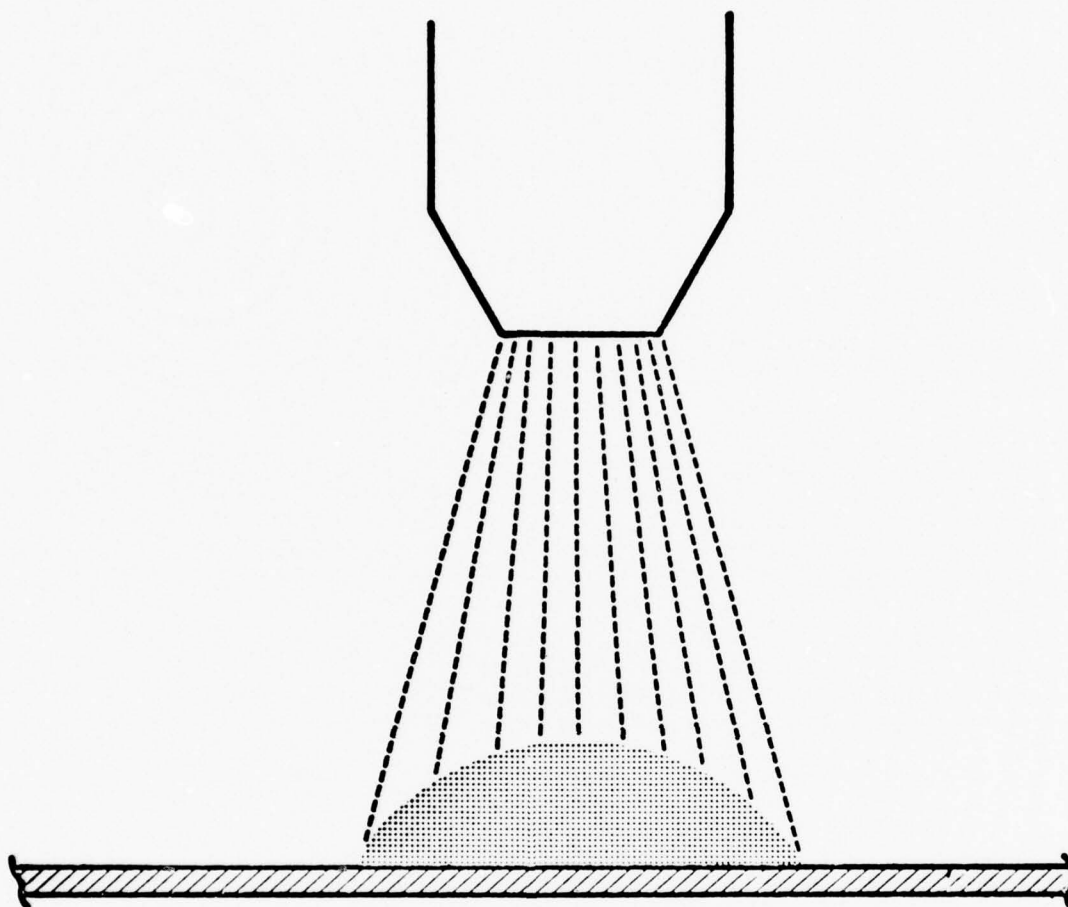


FIGURE 18. TYPICAL CONTOUR OF SPRAY DEPOSIT

The term surface speed of deposition is analogous to the surface cutting speed in machining operations. Specifically, it is the speed at which the surface of the substrate passes the spray gun nozzle and is controlled by the rate of gun movement and either the rotational or sliding movement of the substrate. It is essential that automated or semiautomated tooling be employed to control properly the surface speed of deposition.

A basic criterion that may be used to establish the proper surface speed is that the thickness of the spray material deposited in a single pass should not exceed 0.006 inch. Should the required coating thickness be in the 0.006 to 0.008 inch range, the coating should be applied in a minimum of two passes. An appropriate surface speed of deposition can be calculated from knowledge of the density of the coating material, the recommended rate of deposition in weight/unit time, and the width and thickness of the desired coating.

The two primary concerns in choosing the surface speed of deposition are that the speed be sufficient to prevent overheating of the substrate and/or coating, and that the gun-work distance and the lateral feed of the gun or substrate produce a sprayed deposit flat and free of ridges -- in other words, the proper spray pattern overlap. Figure 19 illustrates the effect of insufficient pass overlap on surface contour. It can readily be seen that insufficient pass overlap, which is greatly magnified in the figure, would result in a lack of finish stock in some areas, although a micrometer reading would not detect this condition.

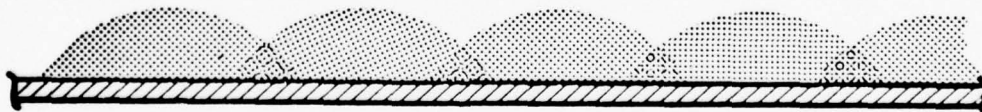


FIGURE 19. INSUFFICIENT PASS OVERLAP

The temperature gradient developed during spraying influences the residual stresses in the coating and substrate. Furthermore, the effective temperature of the substrate affects the time for molten particles to freeze, to react with contaminants in the gas stream, or to react with the substrate. Therefore, it is common practice to provide some means of process temperature control in addition to that characteristic of the other plasma-arc deposition variables employed. Usually, the substrate is preheated before spraying and cooled during the deposition operation.

Substrates are preheated for several purposes. Heating substrates to 250 F will prevent condensation of water vapor in the gaseous environment. More importantly, preheating minimizes the thermal shock of molten particles striking the colder substrate and the residual stresses in coatings and coated components. For the latter reason, preheating is particularly desirable when depositing thicker coatings. The maximum preheating temperature depends mainly on the base material and the temperature rise that will occur during spraying. For instance, high-strength steel workpieces will soften if heated to temperatures above those used for tempering them during heat treatment. High preheating may be undesirable when the differences in thermal expansion coefficients of the coating and substrate are large enough to cause high stresses from cooling between the preheating and room temperature.

It is usually desirable to provide some cooling in order to prevent the temperature rise during processing from exceeding 300 F. Cooling is especially desirable for substrates with low melting points, and for coatings extremely susceptible to oxidation. Thin substrates are often clamped to massive components in order to provide a better heat sink, faster cooling, and lower average process temperatures. Rapid cooling is desirable for some coating materials, molybdenum for instance, because it results in a smaller grain size and higher strength. Cooling is usually provided by directing blasts of compressed, clean, dry air, carbon dioxide, or inert gas at the substrate or at positions just ahead of or behind the plasma stream.

SECTION VI

6. POST-SPRAY FINISHING AND TREATING OPERATIONS

Two features common to all "as-deposited" plasma-sprayed coatings are their surface texture and their porosity. The surface roughness of a typical plasma-sprayed coating measures in the range of 225-450 microinches (AA) -- a fairly rough, abrasive surface. The porosity usually ranges from 3 to 15 percent by volume, depending on the material sprayed and the intended use for the coating. In most cases, plasma-sprayed coatings are applied to machine elements and the coated part must conform to close dimensional tolerances or have a smooth configuration or a particularly accurate shape. In such applications, coatings are often exposed to machine oils or other industrial fluids, which can infiltrate a porous coating, resulting in fluid leakage or corrosion of the substrate. Consequently, many applications require plasma-sprayed coatings to be sealed and surface finished after spraying.

6.1 Sealing

In most thermal sprayed coatings, the porosity, which may range as high as 15 percent by volume, is interconnecting, making the coating pervious to gases or liquids. This permeability can leave the substrate vulnerable to corrosive attack or can result in undesired fluid leakage in certain machine element applications (e.g., a coated piston in a hydraulic cylinder). Furthermore, surface porosity increases the difficulty of producing a smooth finish by machining or grinding.

To prevent corrosive attack or fluid leakage, it is necessary to seal the pores prior to surface finishing. Wax, phenolic, or inorganic sealers are readily available and easily applied. Waxes are useful in preventing infiltration of liquids at low service temperatures, resin-base sealers are effective for service above the boiling point of water to 500 F, and some silicon-base sealers have provided effective protection in salt-spray tests made according to military standards up to 900 F. Epoxy and phenolic sealers are generally effective within the temperature limits of stability.

The most effective method of sealer application is vacuum impregnation, which may be used to fill all accessible porosity. The coated part is immersed in a container of epoxy resin and the container placed in a vacuum chamber. A vacuum is drawn, pulling air from the coating porosity. When the vacuum is released, atmospheric pressure forces sealer into the evacuated pores. Most applications don't require such thorough penetration. Commercially available low-viscosity sealers, which set by an anaerobic reaction

or by heat curing can be brushed or sprayed onto a coated surface at room temperature and are drawn into pores by capillary action. The depth of penetration may be as much as 0.030 in. in some instances. It is important that the sealer penetration be sufficiently deep that it is not removed during the subsequent finishing operation.

Other methods of impregnation are sometimes suitable for filling pores in sprayed coatings to prevent infiltration of air or fluids during service. Roseberry and his colleagues investigated the efficacy of pressure impregnation and a simpler treatment for sealing or filling porosity.⁽⁹⁾ Two metallic coatings, molybdenum and the 80Cr-20Ni alloy, and two oxides, blended Al_2O_3 -13TiO₂ and Cr₂O₃ were used in that work. The coatings, 0.022 and 0.030 in. in thickness, had been plasma sprayed on the following substrates:

1020 steel	K Monel
316 stainless steel	brass
molybdenum	6061-T1 aluminum .

The process parameters used for spraying are recorded by data sheets in Section IX.

Rods for hydraulic cylinders were treated with sealing materials to prevent infiltration of the coatings by air or hydraulic fluid during service. Two types of sealer were evaluated for filling the as-sprayed coatings: a low-viscosity, single-component polyester resin (Loctite 290), and a clear, synthetic phenolic resin (Metco Type BP).

The phenolic sealer was applied to the as-sprayed coatings by immersing the components in a bath of the resin enclosed in a pressure chamber. The vessel was then pressurized with air to 250 psi and held for five minutes. After removal from the chamber, the impregnated components were heated in an oven at 350 F for one hour to cure the phenolic resin.

The polyester resin sealant was applied by brushing the coated shafts as they were rotated in a lathe. Because of its low viscosity, the resin was drawn into the open pores by capillary action. It hardened by an anaerobic process (oxygen not required for curing). After being wetted thoroughly by the polyester, the sealing material was allowed to harden in air and then post-cured by heating for 30 minutes at 250 F.

Based both on performance and on ease of application, the polyester resin sealant (Loctite 290) was selected for use on the coated rods subjected later to functional testing. Visual observation of finished, impregnated surfaces indicated better penetration of the coating, and static pressure tests confirmed these observations. Specifically, when pressurized with hydraulic oil in the bench test apparatus shown in Figure 20, the phenolic-impregnated

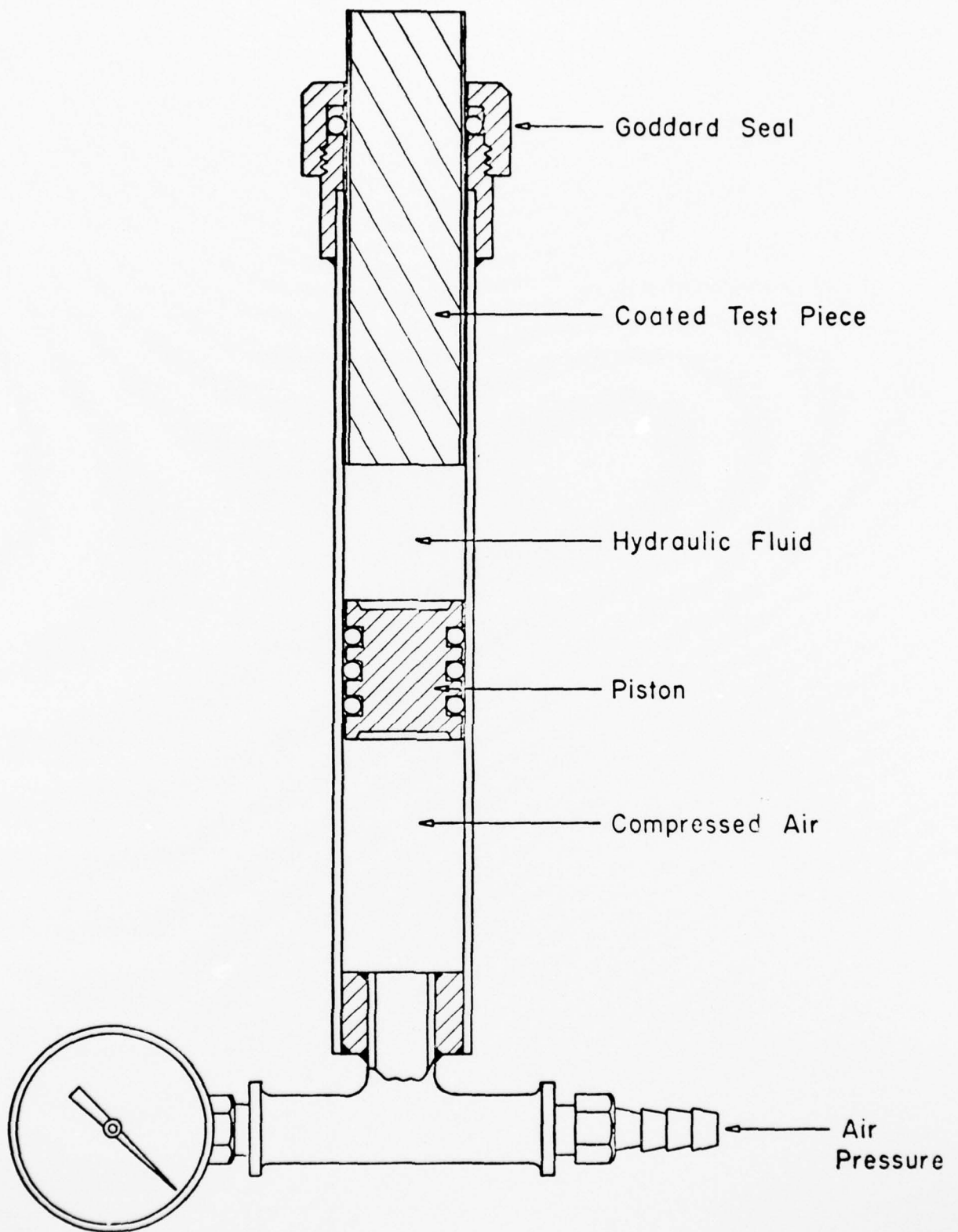


FIGURE 20. PRESSURE-TESTING APPARATUS (9)

shafts permitted infiltration of oil through the coating at pressures as low as 20 psi. On the other hand, polyester-resin impregnated shafts did not leak when pressurized in this apparatus to 100 psi. In later tests, all coated shafts were shown to withstand 2000 psi pressure when they were assembled into the hydraulic wear-test apparatus and allowed to stand under pressure overnight.

Representative coated shafts were subsequently subjected to even greater pressures. The bench-testing apparatus was modified to determine the effectiveness of sealed coatings against air infiltration. Randomly selected shafts representing each of the coating materials were assembled in the apparatus, pressurized with air to 300 psi, and held at pressure for five minutes. The coated surface protruding above the pressurized cylinder was doused with SNOOP leak detection liquid [Mil Spec MIL-L-25567C (ASG) Type 1] and observed. No air leakage was observed in any of the coated rods tested. These shafts were then assembled in a standard hydraulic cylinder and pressurized to 3500 psi with the hydraulic fluid used in the functional wear tests. This pressure was maintained for 15 minutes, during which time no infiltration of fluid through the sealed coatings was observed.

Thermal sprayed coatings usually contain some isolated pores not connected to each other or to the surface. Closed pores cannot be filled by impregnation and may be opened by finish grinding or machining operations. For some applications, it may be desirable to treat spray-coated components with a sealer or filler after machining. This was true for the hydraulic cylinder rods, and epoxy resin worked well for the purpose. Epoxy resin (Epon 828) was applied to the shafts after grinding to final dimensions. The procedure consisted of coating the surface, placing the shaft in a vacuum chamber, scraping the excess epoxy off with a shaft seal, and curing the resin by rotating the shaft in an oven at 150 F for four hours. The presence of the filler in sectioned pores provides some mechanical support to the coating and reduces leakage along the surface. Some materials used for sealing and filling pores or voids in plasma-sprayed coatings are listed in Table

6.2 Surface Finishing

The unique structure of plasma-sprayed deposits requires that considerable care be taken in any machining or grinding operations in order to avoid damage to the coating. Since a sprayed coating is composed of an aggregation of individual particles, improper techniques during the finishing operation could result in "pull-out" of particles, singly or in clusters, thus producing a severely pitted surface. It is essential, therefore, that the sprayed particles be cleanly sheared and not pulled from the surface. It is important to note that even an ideally finished surface will probably not be shiny, but may have a matte finish due to porosity.

TABLE XXIII. DESCRIPTION OF SEALING MATERIALS

Description	Product Identification	Method of Application	Form	Curing Method
Air drying, oil modified phenolic varnish resin	Metco Seal AP	Brush	Liquid	Air dry
Baking type, clear synthetic phenolic	Metco Seal BP	Brush or pressure impregnate	Liquid	Bake at 350 F
Dimethacrylate -- poly-ester resin (anaerobic curing)	Loctite 290	Brush	Liquid	Air dry or bake at 250 F
	Teflon	Pressure impregnate	Liquid	Bake
Phenolic base	Wall Colmonoy ADP	Brush	Liquid	Air dry
Aluminum silicate	Wall Colmonoy SAS	Brush	Liquid	Air dry
Clear vinyl	Wall Colmonoy CVS	Brush	Liquid	Air dry
Aluminum flake, suspended in a vinyl lacquer	Wall Colmonoy AVS	Brush	Liquid	Air dry

The selection of a finishing method depends on the type of coating, its hardness, and its thickness. Consideration should also be given to the properties (hardness, flexibility) of the substrate material. Softer sprayed materials are often finished by machining, especially coatings applied to machine components. Such applications usually involve plasma-sprayed refractory metals or the softer cermets. A good finish can be obtained using high speeds and carbide tools for most such applications. More often, however, plasma-sprayed coatings are finished by grinding.

The physical characteristics of the coating and the substrate influence the selection of grinding equipment and techniques. When done properly, much less stress is imposed on the coating, substrate, and interface in grinding than in a machining operation. Factors to be addressed in finishing by grinding include the type of wheel to be used, as to abrasive medium, e.g., diamond or silicon carbide, abrasive-grit size, wheel hardness and bonding medium; and whether to dry-grind or wet-grind. Some general guidelines regarding these factors provide a starting point, but optimum material removal parameters depend on the material being finished and its intended use.

Generally, wet-grinding is recommended over dry-grinding, provided the proper wheel is used. Sprayed materials, having a different structure than the same materials in solid form, cannot be ground with the same wheels, feeds, speeds, etc., recommended for solid materials. Sprayed metals, in particular, are difficult to grind because they tend to adhere to and "load" a grinding wheel. Consequently, wheels with coarse grain and low bond strength are used to grind metals to prevent loading the wheels.

Guidelines set forth by the American Welding Society (AWS) are helpful in determining a starting point for wheel selection.⁽¹⁰⁾ According to the AWS, if high precision is not required, a 150-300 microinch plasma-sprayed surface may easily be improved to 75-120 microinch using a soft rubber bonded 80-grit silicon carbide abrasive or a vitrified bonded (H-L hardness) silicon carbide stick (80-300 grit) or diamond stick (80-300 grit). Dry grinding at speeds slow enough to avoid overheating is considered appropriate.

For smoother finishes, the AWS states that the best diamond grinding results have been obtained with resinoid-bonded diamond wheels of 50-100 percent concentration, "L" or "N" hardness, with diamond grit sizes of 100 to 400 mesh. Grit sizes 80-120 are used for roughing, 150 to 240 for semi-finishing and 320-500 for high finish.

Wheels with grit sizes appropriate for the surface finishes desired should be used. Results expected from diamond concentrations of 100 are:

Grit Size, mesh	Surface Roughness, microinch (AA)
100	24-36
220	12-18
400	6-12

Flood cooling with a water-base fluid containing a rust inhibitor is recommended.⁽¹⁰⁾ Wheel speeds in the range from 1000 to 6000 feet/minute are considered satisfactory; a harder wheel is preferable for lower speeds. Cross feeds of 0.04 to 0.08 in. and infeeds of 0.0001 to 0.0005 in. are often used.

Smoother finishes than those just mentioned can be obtained by lapping. The achievable surface roughness varies directly with the size of the lapping particles; e.g., lapping with 1-5 microinch diamond grit is expected to produce a surface roughness of 3 microinch (AA).

The coated and sealed rods, described in Paragraph 6.1, required a very smooth surface finish for testing in hydraulic piston wear tests.⁽⁹⁾ In finishing the rods, the surface characteristics of conventional steel cylinder rods supplied by the cylinder manufacturer were taken as the standard to be attained. These standard cylinder rods were specified as having a finished diameter of 1.000 +0.000 -0.001 inch with a 5-10 microinch (AA) surface. "Talysurf" surface roughness measurements indicated that an even better surface finish of 2-3 microinch (AA) could be attained.

To finish the coated rods, they were rotated on centers and ground using a combination of silicon carbide and diamond grinding wheels. Particular attention to technique was required in the case of the $\text{Al}_2\text{O}_3\text{-TiO}_2$ composite and Cr_2O_3 coatings, as the nature of these ceramic materials is such that individual particles within the coating will tend to fracture, or pull out during grinding, making it difficult to obtain a smooth, defect-free surface. Similar difficulties were posed by the molybdenum coatings. Of the four coating materials evaluated, only the 80Ni-20Cr coating yielded a surface finish comparable to the conventional steel cylinder rods.

In grinding the coatings, a No. 120 grit diamond grinding wheel was used for rough grinding and gross stock removal. Finish grinding was accomplished with a No. 180 grit silicon carbide wheel. During finish grinding, stock removal proceeded at a rate of 0.0005 in./pass. The coating thicknesses of 0.022 and 0.030 inch, as sprayed, on different rods were reduced to 0.015 inch by grinding.

SECTION VII

7.

HEALTH AND SAFETY

Plasma spray, like numerous other industrial processes being used today, has its hazards, but those hazards can be eliminated or at least minimized, provided the proper safety precautions are followed. The potential hazards to health and safety of the operators and surrounding personnel can be grouped as follows:

- Dust and Fumes
- Gases
- Arc Radiation
- Noise
- Electrical Shock

These hazards are discussed individually in the following sections. Information given here is intended to supplement and help identify the need for the services of specialists in industrial hygiene and safety.

7.1

DUST AND FUMES

In contrast with other thermal spraying and surfacing operations, the dust and fumes produced during plasma-arc spraying originate primarily with the material being sprayed. Thus, potential dust and fumes hazards can be estimated by considering the hazards associated with the coating material. The magnitude of the hazard presented by dust and fumes depends on the composition and concentration of contaminant in the breathing zone. In cases where control is not certain, air sampling should be accomplished in the spray area to evaluate personnel exposure. The most common method for air sampling is by use of lapel samplers, where the operator carries a unit on his person and air is sampled continuously in the breathing zone. Analysis of samples permits calculation of the contaminant concentration in the operator's breathing air.

Exposures determined by air sampling can be compared to permissible exposure standards established for the contaminant(s) in question. Some commonly used exposure standards and guidelines are available as follows:

<u>Standard</u>	<u>Source</u>
Threshold Limit Values (TLV's)	The American Conference of Governmental Industrial Hygien- ists (ACGIH), revised annually, published in pamphlet form.

Standard	Source
OSHA Standards	The Occupational Safety and Health Administration, U.S. Department of Labor, published in Code of Federal Regulations, Title 29, Part 1910, Subpart Z.
ANSI Standards	The American National Standards Institute.
Hygienic Guide Series	The American Industrial Hygiene Association.
Criteria Documents (Criteria for proposed new OSHA Standards)	The National Institute for Occupational Safety and Health, U.S. Department of Health, Education, and Welfare.

Permissible exposure standards have been subject to frequent change in recent years, as new information on toxic properties develops rapidly. Care should be taken to see that the standards used are up to date.

7.1.1 Ventilation

The principal control measure for airborne contaminants is local ventilation. Existing guidelines for ventilation adequacy are incomplete and their validity is not well documented. Exposure should be monitored by lapel samplers in cases where control adequacy is in doubt.

An exhaust hood is considered necessary in all cases for plasma-arc spraying, general room ventilation being considered inadequate. For the most innocuous spray materials (nuisance particulates -- see the TLV pamphlet referred to above), a freely suspended hood of the type commonly used for welding (attached to flexible hose) may be adequate if spraying is carried out right at the face of the hood and a face velocity (capture velocity) of 200 feet per minute is maintained. The spray velocity must be directed into the hood.

For toxic* materials a totally enclosing hood is specified, with 200 feet per minute face velocity. The hood is to enclose the entire operation, the operator remaining in an upwind position. It is stated further** that an approved supplied-air respiratory protection should be provided when toxic materials are used.

Ventilation design should not overlook adequate make-up to replace air exhausted. Care must be taken to ensure that exhausted air is suitably cleaned and/or exhausted at an appropriate location and in such a manner that problems will not be created and exhaust air will not be drawn into an air intake.

7.1.2 Respiratory Protection

Respirators are occasionally recommended as a control measure, but are in general much inferior to good ventilation control. Where respirators are used, good work practice standards must be followed to avoid losing the protection afforded by the respirators. Chief among standards is the OSHA Respiratory Protection standard 1910.134, which is supplemented by American National Standard Practices for Respiratory Protection Z88.2. A few pivotal requirements of these standards are:

- Approved*** respirators are to be used.
- Respirators are to be selected on the basis of hazards to which the worker is exposed (even a respirator approved for a given material will be unacceptable if the concentration of air contaminant is too high).
- Users are to be instructed and trained in proper use and limitations of the respirators.
- Respirators are to be properly cleaned, stored, maintained, and inspected on a routine basis.
- Appropriate continuing surveillance of the respirator usage is to be maintained by responsible qualified persons.

* Precisely what materials belong in this category has not been defined clearly, but it has been suggested that all materials with exposure limits lower than 10 milligrams per cubic meter or 100 parts per million be included. At the present time cadmium, cobalt, lead, copper, zinc, chromium, and nickel would be included in addition to highly toxic materials like beryllium.

** Industrial Ventilation A Manual of Recommended Practice, 13th Edition, Print No. VS 415, American Conference of Governmental Industrial Hygienists.

*** Approval is by the National Institute for Occupational Safety and Health (NIOSH); Bureau of Mines approvals are being phased out.

The plasma-arc spraying environment is also characterized by the presence of various gases, some of which may present a hazard to the health of equipment operators. In addition to the gases used to form the plasma and propel the material being sprayed (argon, helium, nitrogen, hydrogen, etc.), the following gases may be present:

- (1) Nitrogen Oxides. Various oxides of nitrogen may be produced during spraying by reactions between atmospheric nitrogen and oxygen. Nitrogen dioxide (NO_2) is of most concern.
- (2) Ozone. Ozone may be produced by the reaction of atmospheric oxygen and ultraviolet radiation from the plasma arc.

Other gases may be present under certain conditions. For example, phosgene and dichloroacetyl chloride can be produced by reactions in the presence of ultraviolet radiation of the chlorinated hydrocarbons (e.g., trichloroethylene) used for degreasing metal surfaces. Such reactions may occur if spraying is done near a degreasing facility or in an area where vapors from the degreasing facility can be carried by natural or artificial air currents. Both of these gases are classified as pulmonary irritants; also, in addition to being extremely toxic, phosgene can cause dermatitis upon contact with the skin. The toxicity of dichloroacetyl chloride is not well defined and the threshold limit value has not yet been established for this gas.

As in the case of fumes, the hazards presented by gases are a function of the type of gas and its concentration in the breathing zone. The gases used for plasma-arc spraying are not toxic; however, a deficiency of oxygen can occur if they are leaked into a confined area and the plasma gases displace oxygen in the atmosphere. Hydrogen is, of course, a highly flammable gas and must be handled accordingly. Nitrogen dioxide and ozone are very toxic, and problems can be created if these gases are present. Gas contaminant concentrations are expressed in part of gas per million parts of air (ppm); the threshold limit values (TLV's) for ozone and nitrogen dioxide are 0.1 and 5 ppm, respectively. The nitrogen dioxide TLV is a ceiling value which should not be exceeded even for a portion of the working day.

Few problems with gases should be experienced if ventilation is adequate in accordance with the guidelines discussed previously under Dust and Fumes. Spraying facilities should not be located near those used for degreasing or other operations using chlorinated solvents.

7.3 ARC RADIATION

Ultraviolet, visible, and infrared radiation are produced by plasma-arc spraying operations. As shown below, the spectrum of the plasma arc can be divided according to wavelength:

<u>Type of Radiation</u>	<u>Wavelength, nm*</u>
Extreme ultraviolet	4-200
Ultraviolet	200-400
Visible	400-750
Infrared	750-1300
Far infrared	1300-greater

Of course, the boundaries of these regions are not as well defined as this table indicates. From the health and safety standpoint, radiation with wavelengths of 200 to 1300 nm is of most concern. Also of concern is the radiation intensity, a parameter whose magnitude is a function of (1) the spraying process, (2) the plasma and propellant gases, (3) the substrate, (4) the operating variables, and (5) the distance from the plasma arc.

The problems associated with each type of radiation are reviewed in the following sections.

7.3.1 Ultraviolet Radiation

The radiation of ultraviolet energy by the plasma arc can have both a direct and an indirect effect on the health and safety of equipment operators. Directly, exposure to ultraviolet can cause temporary or permanent damage to the eyes, and it can affect the skin in the same manner as solar radiation. Ultraviolet is the portion of sunlight believed responsible for excess skin cancer among outdoor workers. Indirectly, toxic gases can be produced by reactions between ultraviolet radiation and atmospheric oxygen, nitrogen, or chlorinated hydrocarbons. Since such reactions have already been reviewed, this discussion is concerned with the direct effects of ultraviolet radiation.

As noted previously, ultraviolet radiation can produce temporary or permanent eye damage. However, the most common eye ailment experienced by equipment operators is "arc-flash" or "arc-eye". Both of these terms are misnomers for conjunctivitis, an inflammation of the transparent membrane that covers the inner surface of the eyelids and the white of the eye. This is a temporary condition from which recovery is complete unless re-exposure occurs. Conjunctivitis can be prevented by the use of proper filter lenses. It is recommended that at least a shade 12 should be used for any close viewing of the plasma. Eye protection should also be furnished to nearby workers who may be exposed to arc radiation.

* Wavelength is measured in nanometers (nm) or 0.000,000,001 meter.

Ultraviolet radiation can also produce a skin burn that is similar to sunburn and just as painful. Such burns can occur on unprotected parts of the body that are exposed to arc radiation during spraying. They can be prevented by wearing protective garments. Heavy work clothing is advisable for all but the mildest exposure, and aluminized material should be used for heavy exposure.

It should be noted that eye damage and skin burns can also be caused by ultraviolet radiation that is reflected by walls and ceiling of spraying enclosures. Such surfaces can be coated with paints that absorb rather than reflect radiation.

7.3.2 Visible Radiation

There is little evidence that visible radiation constitutes a serious hazard in plasma-arc spraying. However, glare can produce eye discomfort, fatigue, and headache. The use of the correct filter lenses provides adequate protection against visible radiation.

7.3.3 Infrared Radiation

Most of the radiation from the plasma arc lies in the infrared region. Excessive exposure to this type of radiation can produce retinal burns and cataracts, which can be prevented by the use of filter lenses that absorb infrared radiation.

Infrared radiation also produces a sense of heat on the skin. Heat produces physiological as well as psychological effects on equipment operators; the physiological effects are most important from the health and safety standpoint. Excessive heat can produce discomfort, sweating, fatigue, and headache; in addition, performance is impaired. The psychological effects of heat are more difficult to measure. However, there is little doubt that efficiency is decreased.

The use of adequate protective clothing and standard types of eye protection for ultraviolet will also serve to minimize exposure to infrared radiation.

7.4 NOISE

The noise associated with plasma-arc operations may present a hazard because of its intensity and frequency. Noise levels in excess of 90 dB(A) have been measured near plasma arcs. Also, the noise produced by such arcs is distributed over a wide frequency range, including the high frequency range to which the ear is more sensitive. The effect of noise on hearing is a function of the sound intensity, pitch or frequency, and duration.

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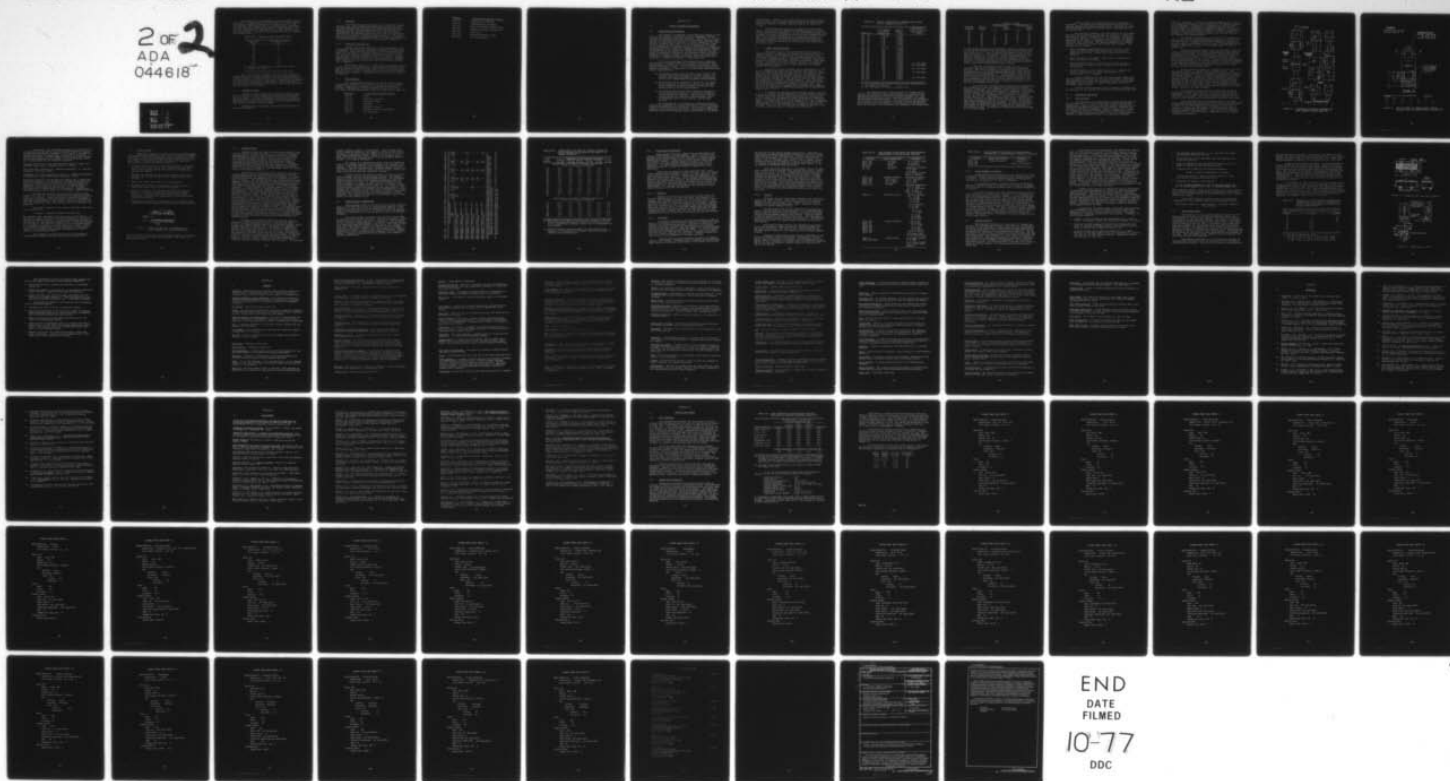
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Threshold limit noise-exposure values are given in Table XXIV. In Table XXIV, the sound level is in decibels weighted according to a standard A-weighting curve, which de-emphasizes low-frequency and very-high-frequency sounds. Sounds in the 1000 to 6000 Hz range are less important either because they are less important to the ear (low frequencies) or less important in the workplace (high frequencies). A-weighting is incorporated in the design of most sound level meters.

TABLE XXIV. THRESHOLD LIMIT VALUES

Sound Level, dB(A)	Duration hours
80	10
85	5
90	2
95	1
100	0.5
105	0.25
110	0.125
115	0.0625

No exposure to continuous or intermittent noise in excess of 115 dB(A)

The noise control equipment that is recommended by the equipment supplier should be used when exposures threaten to exceed the TLV's, and the TLV's should be supplemented by ear muffs or ear plugs. When exposures reach sound levels approaching or exceeding the TLV's, initial and periodic hearing tests to help identify hearing problems and sensitive individuals.

7.5 ELECTRICAL SHOCK

Electrical shock hazards from plasma equipment are similar to those presented by many types of electrical equipment. Only well-designed and well-maintained equipment should be used, and it must be properly insulated. The open-circuit, operating, and supply circuit voltages must be high enough to present a serious hazard.

Operators of the equipment are protected by two control measures:

7.5.1 Grounding

To protect against the possibility of equipment cabinets, housings, etc. being accidentally energized by short circuits, all such exposed metal parts should be connected together and to electrical ground. The grounding conductors used should be as prescribed by the National Electrical Code. The Code usually requires that the grounding path be carried back to a panel where the circuit originates. Operators should be alert to the importance of maintaining the grounding system and reporting immediately any suspected damage to conduit or other grounding conductor.

7.5.2 Insulation and Guarding

Cables, electrodes, and other circuit parts which carry a hazardous voltage must be insulated or guarded to prevent operator contact. When installed according to the National Electrical Code, all live parts or equipment operating at 50 volts or more will be guarded against accidental contact by persons or conductive objects. Operators should be alert to see that all guards, access panels, etc. are maintained in proper position. Insulated parts should be inspected before each use and withdrawn from service if damaged.

Maintenance, repairs, or modifications of equipment should be done only by qualified persons. Additional guidance with respect to electrical hazards can be found in American National Standard Z49.1-1973, Safety in Welding and Cutting, and in the American Welding Society pamphlet, "Recommended Safe Practices for Plasma Arc Cutting".

7.6 OSHA STANDARDS

The following OSHA Standards for general industry apply to plasma-arc spraying and related activities, either as legally binding regulations or as guidance in good health and safety practice. OSHA General Industry Standards can be found in the U.S. Code of Federal Regulations, Title 29, Chapter XVII, Part 1910.

<u>Standard</u>	<u>Title and/or Subject Covered</u>
1910.95	Noise
1910.96	Radiation (Ionizing)
1910.101	Compressed Gases
1910.103	Hydrogen
1910.106	Flammable Liquids
1910.132	Personal Protective Equipment
1910.133	Eye Protection

<u>Standard</u>	<u>Title and/or Subject Covered</u>
1910.134	Respiratory Protection
1910.166	Compressed Gas Cylinders
1910.242	Hand and Portable Powered Tools
1910.252	Welding, Cutting, and Brazing
1910.308	Electrical
1910.309	National Electrical Code
1910.1000	Air Contaminants

SECTION VIII

8. QUALITY ASSURANCE PROCEDURES

8.1 QUALIFICATION PROCEDURES

The success of plasma-arc coated components depends on the skill of the operator, the condition of the equipment, and the selection of appropriate process variables. Therefore, it is logical that qualification tests be part of the quality assurance procedures for organizations required to produce components for severe service. Although there seem to be no generally accepted industrial standards for the purpose, the major purchasers of plasma-sprayed components for aircraft engines require potential suppliers to demonstrate their capabilities before being approved as vendors. The qualification procedures are intended to establish that the vendor has operators, equipment, and processes capable of thermal-spraying acceptable coatings, of specific types, on appropriate substrates. Somewhat similar qualification procedures may be of interest, or possible usefulness, in Navy facilities.

So far as plasma-spray system operators are concerned, qualification procedures should be intended to demonstrate skill and knowledge, the ability to follow process instructions, and to produce acceptable products. As in the widely used welding qualification procedures, a combination of the following exercises would seem to be appropriate.

- The operator should take and pass a short written test covering questions pertinent to cleaning, surface preparation and masking procedures, and the general principles of the plasma-spraying equipment and procedures.
- The operator should demonstrate familiarity with appropriate equipment by connecting, setting up, and operating plasma-arc spraying equipment according to the manuals supplied by the manufacturers.
- The operator should demonstrate capability by depositing an acceptable coating to a specified thickness on an appropriate test specimen. The quality of the test coating should be judged by suitable methods -- some are described later in this section, such as bond strength.

The qualification of equipment for plasma-arc spraying required by purchasers of critical components is usually directed toward two quality-control objectives. First, using a qualified operator, the equipment must be shown to be capable of depositing coatings meeting the acceptance quality agreed upon by the vendor

and purchaser. Secondly, all of the metering and control devices governing deposition variables must be shown to be properly calibrated and checked at regular intervals such as at least every 30 days.

The purchaser of high quality plasma-arc coated parts usually requires that the deposition process proposed for production be qualified by experiments. In the qualification tests, the coatings of interest are deposited on test strips representing the material and surface characteristics of those to be used in the shop. The coatings are deposited to specified thicknesses by qualified operators using written process sheets and qualified equipment. Then the coatings are evaluated by test methods mutually agreed upon.

8.2 POWDER CHARACTERIZATION

Suppliers of shot, grit, and powders of interest for thermal spraying or substrate preparation ordinarily provide the chemical analysis and information on the particle size of the materials they offer for sale. When chemical compositions are to be checked, standard analytical methods are employed. Most metal samples are analyzed by optical emission spectroscopy or wet techniques. Gas contents of metal powders, coatings, or substrates are determined by vacuum fusion. Other materials are usually analyzed by wet methods.

The particle sizes of powders can be determined by a variety of procedures and described by different designations. For particle sizes larger than 325 mesh (45 μm), the range is characterized by the minimum and maximum cumulative percentages, by weight, that will pass through or be retained by screens (sieves) with different designation. That system is used for most particulate materials of interest in plasma spraying. Table XXV gives the designations and openings of wire-cloth sieves used for testing or classifying materials according to particle size. The sieves to be used for that purpose and the methods for conducting the tests are described in ASTM Specifications E11-70 and B214-66, respectively. (25,26)

In general the apparatus used for sieve analyses consists of a set of standard sieves assembled in suitable order by nesting, a mechanical shaker, and an analytical balance for weighing the original sample and the sample of powder retained on a particular sieve. The balance should be able to weigh a 100-gram (0.2205 lb) sample to a sensitivity of 0.01 gram (0.002205 lb). The recommended sample weights for sieve analyses are 100 grams (0.2205 lb) and 50 grams (0.1102 lb) for materials having apparent densities, respectively, above or below 1.5 grams/cm^3 (93.64 lb/ft^3).

TABLE XXV. NOMINAL DIMENSIONS FOR STANDARD TEST SIEVES
(U.S.A. Standard Series) (25)

ASTM Sieve	U.S. Standard and ISO (a) Designation	Nominal Opening, inch	Nonstandard Tyler Sieves and Designation, μm (b)
No. 8	2.36 mm	0.0937	
No. 10	2.00	0.0787	
No. 12	1.70	0.0661	
No. 14	1.40	0.0555	
No. 16	1.18	0.0469	
No. 18	1.00	0.0394	
No. 20	850 μm	0.0331	
No. 25	710	0.0278	
No. 30	600	0.0234	
No. 35	500	0.0197	
No. 40	425	0.0165	
No. 45	355	0.0139	
No. 50	300	0.0117	
No. 60	250	0.0098	
No. 70	212	0.0083	
No. 80	180	0.0070	175 (80 mesh)
No. 100	150	0.0059	149 (100 mesh)
No. 120	125	0.0049	
No. 140	106	0.0041	104 (150 mesh)
No. 170	90	0.0035	
No. 200	75	0.0029	74 (200 mesh)
No. 230	63	0.0025	
No. 270	53	0.0021	
No. 325	45	0.0017	44 (325 mesh)
No. 400	38	0.0015	

(a) ISO (International Standards Organization) designations in millionths of a meter.

(b) μm = millionth of a meter = 0.000040 inch.

Data obtained from a sieve analysis of a powder should identify the specific weight fractions passing a particular size opening and retained on screens with smaller openings. (26) The classification should be in size steps small enough to be useful for characterizing the material. It is customary to use successive sieves covering the size range of interest, or every second sieve. For instance, the sieve analysis of a powder for plasma spraying might be recorded in the following form (exemplary, not actual data).

Retained on Sieve	Passing Sieve	Weight, percent			
		Material A		Material B	
			Cumu- lative		Cumu- lative
30		0		0	
35	30	3	3	21	21
40	35	7	10	21	42
45	40	11	21	21	63
50	45	16	37	16	79
60	50	21	58	11	90
70	60	21	79	7	97
80	70	21	100	3	100
	80				

For the assumed distributions indicated, the median particle size is smaller for material A and the proportions of particles larger than sieve size No. 50 are 37 percent for Material A and 79 percent for Material B. The example indicates that characterizing a powder as being -30 + 80 mesh is a rather gross description. It may mean comparatively little for control purposes unless the material in question is known to have been produced by the same supplier by consistent procedures and to have performed satisfactorily in previous plasma-spraying applications.

Particle sizes smaller than 325 mesh (0.0017 in.) are used when very smooth finishes must be produced by plasma-arc spraying. When it is necessary to determine their size and size distribution, the elutriation methods described in ASTM Specification B293-60 are applicable.⁽²⁷⁾ Those methods, based on the velocity of particles falling in a countercurrent of air or gas, are not strictly accurate for porous or nonporous powders, but give reproducible results even on those types of particles. The method is appropriate for particles ranging in size from -5 to over 40 μm (-0.0002/+0.0016 inch).

For scientific investigations, particle size distributions and information on particle shape can be obtained by two more elaborate procedures. The equipment used is known as the Coulter Counter and the MSA (Mine Safety Appliance) apparatus. The Coulter Counter measures the change in electrical resistivity across an orifice as particles, suspended in an electrolyte, are passed through the orifice. This change in resistivity is related to the volume of the particle passing through the orifice. This instrument has an effective range of 1.0 to 500 microns. The MSA is a centrifugal-sedimentation type equipment. It makes use of centrifugal force in speeding the settling time of very fine particles and extends the practical range of sedimentation techniques to the submicron range. The MSA has an effective particle size range of 0.1 to 80 microns.

Particle shape can be obtained by use of microscopic techniques. A minimum of 500 particles should be measured. The diameter maximum and diameter minimum ratio is obtained and, using the techniques described by Irani and Collis, the shape can be calculated.(28)

For practical purposes, the "true" density of most powder particles can be measured by dividing the weight of a sample by the amount of water it displaces.(29) If the powder is representative of the material in the coating after spraying, the density determination is useful in computing the porosity of the sprayed coating by some techniques. The density measurement of powders requires an analytical balance and a clean 50-ml graduate. The procedure is as follows:

1. Fill the graduate approximately half full of water. Use a funnel to avoid wetting the inside wall of the graduate above the water level. Record the volume of water in the graduate.
2. Weigh a sample of dry powder, either new or sprayed material, and record the weight.
3. Drop the powder sample in the water and record the water volume after the sample settles, reading at the bottom of the meniscus.
4. Divide the weight of the powder sample by the apparent increase in water volume to obtain the density; e.g.,
 $45.1 \text{ g}/10 \text{ ml} = 4.51 = \text{density}.$

Useful information on flow rates and apparent densities of powders can be obtained with the Hall Flow Funnel. These data give information as to the ease of handling of the powder during processing, and the density to expect when powder is placed in the powder feeding device.

Precise information about the true density of powders can be obtained with the Micromeritics Helium-Air Pycnometer Model 1302.

8.3 MECHANICAL PROPERTIES

8.3.1 Bond Strength

The strength of the bond between a plasma-sprayed coating and the substrate is of vital importance for most applications. Consequently, tensile testing is the most commonly used method for evaluating the mechanical properties of coatings. Approved procedures for determining bonding or adhesive strength of a coating to a substrate, and of the coating itself, are described in the national standard designated as ASTM C633-69.(30) That method is limited to flame-sprayed coatings with thicknesses exceeding

0.015 in. (0.38 mm). The tests are usually conducted at or near room temperature because of the properties of the adhesives applied to the specimens. The method is recommended for qualification, quality control, and component or process acceptance testing. It is also useful for comparing the adhesive or cohesive strengths of different coatings or different methods of substrate preparation. Because of complicating factors, however, the strength data are not suitable for design purposes.

The standard bond-strength test consists of thermal-spray coating one face of a substrate fixture, bonding the coating to the flat face of a loading fixture with an adhesive, and then applying a tensile load normal to the coating. The ASTM publication should be consulted, but many of the recommendations follow in summary form.

The tension testing machine used for determining bond and coating strengths should be capable of determining loads within a variation of 1 percent. It is desirable to increase the load at a constant rate of cross head travel between 0.030 and 0.050 in./min (0.013 and 0.021 mm/s). It is essential that the load be applied perpendicular to the coating. If the self-aligning devices on the testing machine do not prevent eccentric loading and bending of the specimen, apparatus of the type shown in Figure 21 should be used. That figure also shows a method of connecting the self-aligning fixture to a test specimen.

A test specimen consists of a substrate fixture, to which the sprayed coating is applied, and a loading fixture. Both fixtures should be round solid cylinders not substantially shorter than their diameters. Figure 22 shows the dimensions of fixtures, for either loading or substrate coating, considered suitable for tensile testing. One end of each fixture should be capable of attaching to the self-aligning shackles of the testing machine. Both ends of each fixture shall have facings parallel to each other and normal to the loading axis. Facing diameters of 0.9 in. (23 mm) to 1.0 in. (25 mm) are appropriate and should be the same, within 5 percent, for both fixtures.

The substrate fixture should be constructed of metal, preferably the same metal that will be used for the production substrates. If not otherwise specified, the fixture can be made from 1018 or 1020 steel. After use, substrate fixtures can be ground, given suitable surface preparation treatments, and reused.

Some organizations use nonstandard tensile specimens patterned after the ASTM configuration for measuring bond strengths. A convenient variation employs a test button or disk, 0.200 to 0.250 in. (5.08 to 6.35 mm) thick. The button is ordinarily similar in composition and hardness to the production part and given the same surface preparation. Only one face is coated by thermal spraying. The test disk is cemented to the two loading fixtures by an adhesive.

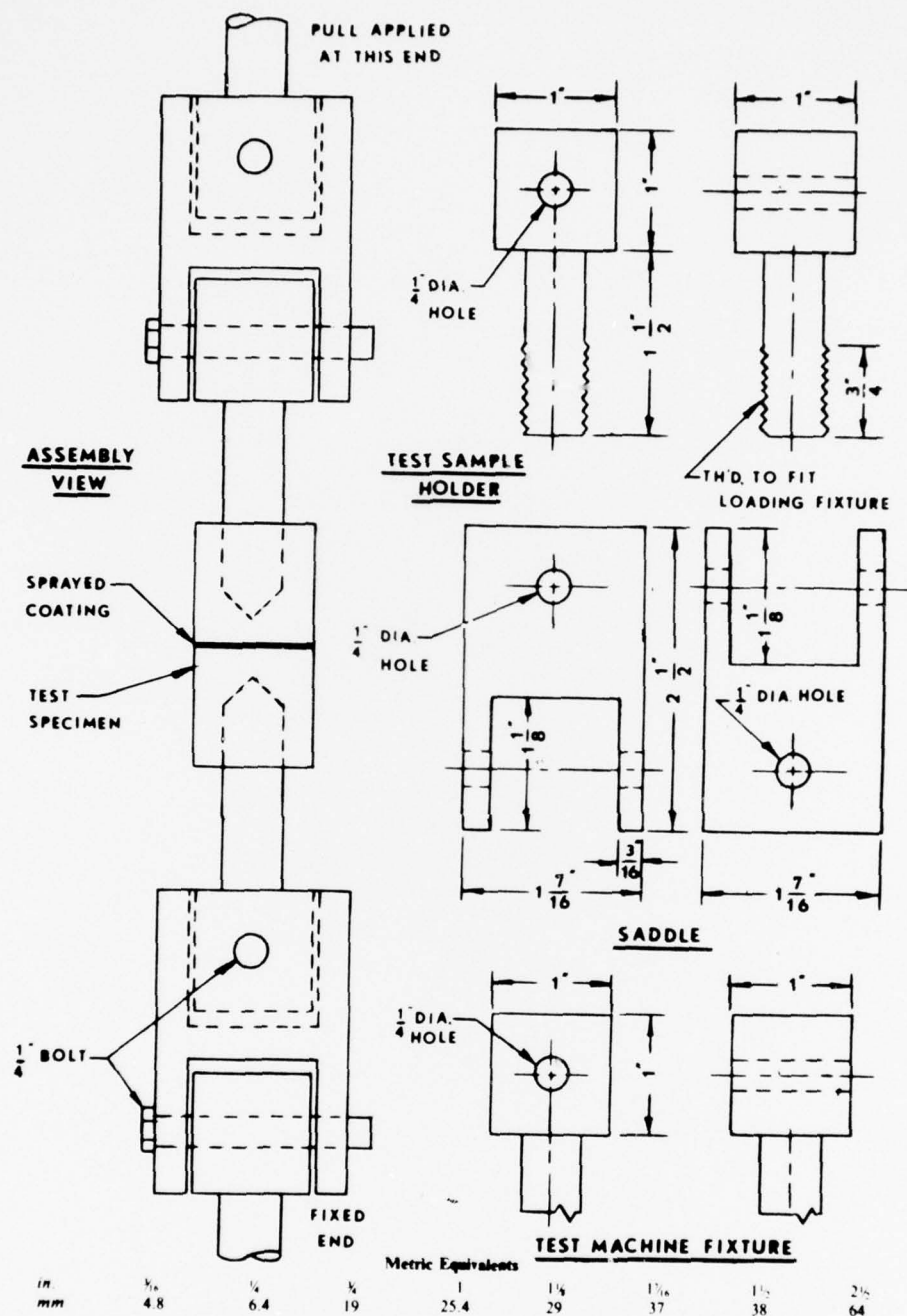


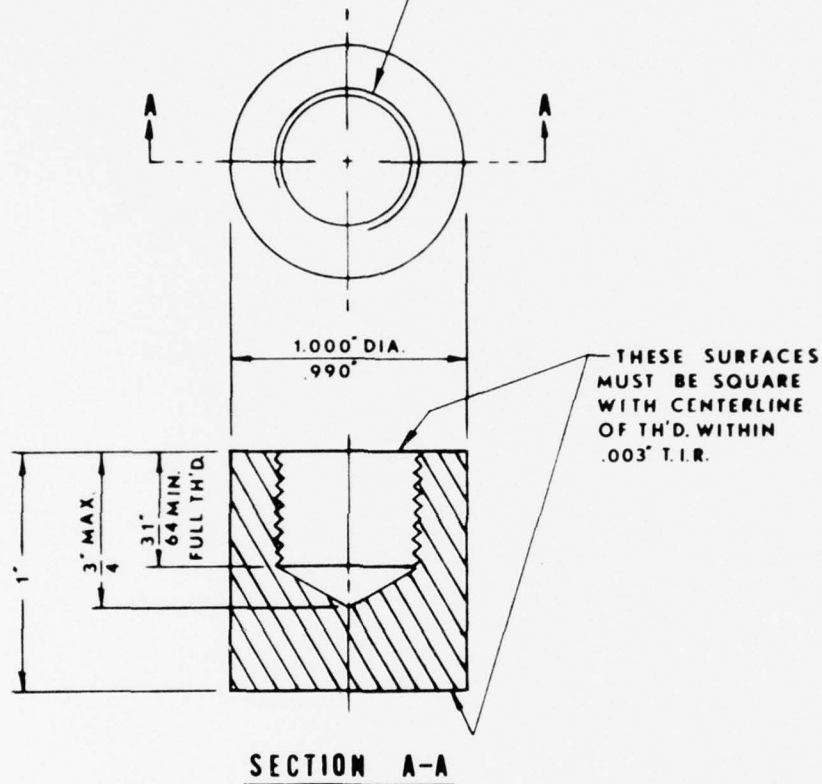
FIGURE 21. FIXTURES FOR ALIGNING SPECIMENS FOR BOND STRENGTH TENSILE TESTS (30)

TOLERANCE

FRACTIONS - $\frac{1}{64}''$ T.I.R. - .003"
MACHINED SURFACES $\frac{125}{\sqrt{\text{RMS}}}$

SUGGESTED TH'D. SIZE

(MIN.) $\frac{1}{2}''$ - 20 UNF - 2B TH'D.
 $\frac{5}{8}''$ - 18 UNF - 2B TH'D.
(MAX.) $\frac{3}{4}''$ - 16 UNF - 2B TH'D.



MATERIAL: 1" DIA. x 1" LG. - PER SPECIFICATION

24" = 1' - 0"

U.S.	0.003 in.	$\frac{1}{32}$ in.	$\frac{1}{16}$ in.	$\frac{1}{8}$ in.	$\frac{1}{4}$ in.	
Metric	0.08 mm	0.39 mm	12.3 mm	12.7 mm	15.9 mm	
U.S.	$\frac{1}{8}$ in.	0.990 in.	1 in.	1.000 in.	1 ft.	24 in.
Metric	19 mm	25.15 mm	25.4 mm	25.4 mm	0.3 m	610 mm

FIGURE 22. TEST SPECIMENS FOR ADHESION AND COHESION STRENGTH TESTS ON THERMAL-SPRAYED COATINGS (30)

The material used for adhesive bonding the coating to the loading fixture must have a tensile strength at least as high as the adhesive and cohesive strengths of the coating. The adhesive bonding agent shall be sufficiently viscous not to penetrate through the coating. Several commercial resins that cure and harden at room temperature are suitable. Among those known to have given satisfaction are the following materials:

Conapoxy AD-1222 resin with Conacure EA-03 catalyst (Conap, Inc., 184 East Union Street, Allegheny, New York 14706).

Epon 911F (Shell Chemical Co., Adhesives Department, P.O. Box 831, Pittsburg, California 94565).

Bondmaster M 777 (Pittsburgh Plate Glass Co., Adhesives Department, 225 Belleville Avenue, Bloomfield, New Jersey 07003).

Brochures provided by the manufacturers explain how to apply and cure their adhesives. The strength of the adhesive bonding agent shall be determined each time the bond or coating strength is determined. This should be done by using the bonding agent to attach two loading fixtures and testing, in order to detect deterioration or improper mixing of the adhesive. Depending on the bonding agent, the strength is of the order of 4000 to 8000 psi.

The ASTM testing specification requires coating thicknesses over 0.015 in. (0.38 mm) and uniform in thickness to within 0.001 in. (0.025 mm). Larger variations in thickness should be corrected by machining or grinding before testing. The number of specimens tested depends on the purpose to which the data will be put. For acceptance tests, data from at least five specimens are desirable.

The bonding strength or the cohesive strength of the coating is taken to be:

$$\text{Strength} = \text{Maximum Load/Cross-Sectional Area} .$$

The strength value represents the weakest part of the system. If the failure occurs entirely at the coating-substrate, the value is reported as adhesion strength. If the failure occurs entirely in the coating, the strength is considered to be the cohesive strength of the coating. The location of the failure is often determined by a low-power microscope at a magnification up to 100X. Failure in the adhesive bonding agent can be considered a satisfactory result if the strength value exceeds requirements for quality assurance or qualification tests.

Other methods of determining the tensile strengths of thermal-spray coatings have been described in brochures. (29)

8.3.2 Shear Strength

Methods for judging the shear strength of plasma-sprayed coatings have been developed by suppliers of equipment and powders. A test that is used quite frequently consists of coating a half-inch band of a solid round cylinder and then applying axial pressure to shear the coating from the cylinder. The supplier's instructions on testing procedures follow.⁽²⁹⁾

1. Make two sleeves to fit a 1-inch round specimen as indicated in Figure 23. Drill and tap each sleeve to take one set screw. The equipment avoids the necessity for machining or grinding the coating.
2. Assemble the cylinder and sleeves and abrasive blast both the bare area of the cylinder and the tapered ends of the sleeves.
3. Remove the sleeves and remove any loose abrasive.
4. Reassemble the sleeves on the specimen, tighten the set screws and preheat as for production spraying.
5. Apply the coating on the specimen and sleeve as shown in Figure 23. Friable or porous coatings should be given a top coat of stainless steel (Metco 420) or Ni-Cr alloy (Metco 43C) to improve the distribution of the shear stress over the test section.
6. Loosen both set screws and measure the shear strength, using a laboratory machine capable of measuring the load accurately.

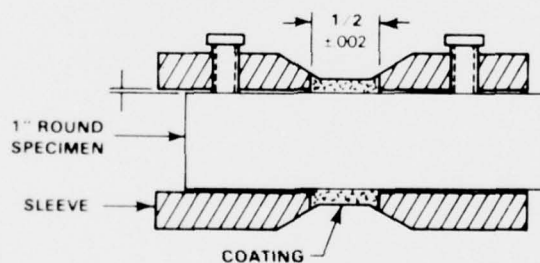


FIGURE 23. ROUND SPECIMEN USED TO DETERMINE THE SHEAR STRENGTH OF A SPRAYED COATING⁽²⁹⁾

The test description mentions that shrinkage stresses in the coatings will affect the measured shear strength, very likely to a considerable extent.

8.3.3 Hardness Tests

Hardness values are often mentioned when comparing coatings and effects of spray variables. It is usually a misuse of hardness data to consider them as measures of coating strength. The measurements may be of more value for quality control and for evaluating process variables. Because most coatings are thin, porous, and characterized by relatively low cohesive strengths, macrohardness tests are not applicable. Although microhardness data are more useful, they require careful determinations and interpretations. Rockwell Superficial Hardness tests are simpler to conduct and are considered useful for quality control and processing studies.

Specimens for microhardness tests should be polished to a metallographic-quality finish because the dimensions of the hardness impressions are measured with a microscope. The measuring microscope shall be graduated in 0.5- μm (0.00002-in.) divisions or smaller. The standard methods for determining the microhardness of materials are described in ASTM standard E384-73.⁽³¹⁾ That publication gives the procedures for determining microhardness numbers using both Knoop and Vickers (or Diamond Pyramid) indentors. In both systems, the hardness number is the pressure exerted on the specimen by the diamond indenter used to produce the impression. The indentors used for Knoop and Vickers determinations are of different shapes and the pressures are calculated for different areas. On the Knoop scale, the pressure is the load in kgf (2.2 lb-force) divided by the projected area of the impression in mm^2 . The pressure or hardness number on the Vickers scale is calculated from the contact area of the indentation by assuming that it is the imprint of an undeformed indenter. In principle, both systems of measuring microhardness are less affected by porosity than one based on measuring the indenter travel caused by a specific increase in load. Microhardness tests are usually made transverse to the coating surface, even though service loads are usually normal to the surface and hardness may vary because of microstructural anisotropy. It is often convenient to make microhardness measurements on specimens prepared for metallographic studies of coating and substrate microstructures and coating porosity.

Procedures for Rockwell Superficial Hardness tests are described in ASTM standard E-18-74.⁽³²⁾ They are far better suited to evaluating sprayed coatings than standard Rockwell tests because they employ smaller loads. The superficial hardness instrument measures the difference in the depths of indentation caused by the minor load (3 kgf or 6.72 lb-force) applied first and the major load which may be 15, 30, or 45 kgf. One unit on the superficial hardness scale represents a penetrator movement of 0.001 mm (0.00004 in.) between minor and major loads. Of the five types of indentors used for Rockwell superficial hardness determinations, the most commonly used are the diamond penetrator (N scale) and the 1/16-inch (1.588 mm) ball (T scale). In reporting

Rockwell hardness numbers, the penetrator, load, and dial used should always be indicated. For example, a value of "81 HR 30 N" indicates a superficial hardness number of 81 on the 30 N (30 kg load, diamond penetrator) scale. There is no reliable general method of converting hardness numbers from one Rockwell scale to another, or to tensile strengths.

The choice of an appropriate scale to use for measuring superficial hardness depends on the hardness and thickness of the coating. Table XXVI provides some information for guiding such judgments. When the substrate has a hardness somewhat similar to that of the coating, and the coating is thick enough, heavier test loads give more reliable and consistent readings.

Several precautions should be taken in conducting superficial hardness tests on plasma-sprayed coatings. The surface should be clean and free of gross imperfections. The impressions should be spaced at least three impression-diameters from each other and from a free edge. At least five determinations are usually necessary to obtain reliable results. A surface finish of 30 microinch is recommended for using the 15 N scale; slightly rougher surface finishes are adequate for the other scales. Flat surfaces are preferable for hardness tests; measurements on curved surfaces give erroneous values. The factors listed in Table XXVII should be used to correct hardness values determined on round specimens. The corrections vary with the curvature, type of penetrator, and the hardness value.

8.4 METALLOGRAPHIC EXAMINATIONS

Metallography is one of the prime tools used by materials people to evaluate the quality of flame-sprayed coatings. Since approval of a coating for a given application depends on the results of metallographic study, it is imperative that the specimen studied be representative of the coating, and that it be properly prepared.

The metallographic procedures are similar to those used for preparing most wrought materials. However, specimens of flame-sprayed coatings require some special techniques. The structure of the coatings is nonhomogeneous, consisting of particles mechanically and metallurgically bonded to each other. The coatings contain oxides and metallic phases not normally found in wrought materials. Care must be taken in the sectioning and polishing to avoid pulling them out, thus avoiding false evaluations. Proper preparation is vital to preserve the integrity of the microconstituents. Ultrasonic cleaning should be avoided, and vibratory polishing used rarely.

TABLE XXVI. GUIDE FOR SELECTING SCALES FOR ROCKWELL SUPERFICIAL HARDNESS TESTS (32)

	Coating Thickness (a)									
	Inch									
	mm	0.006	0.010	0.014	0.018	0.022	0.026			
		0.15	0.25	0.36	0.46	0.56	0.66			
Approximate Hardness, Rockwell C (b)		65	55	32	-	-	-			
Dial Reading, 15 N Scale		92	88	76	-	-	-			
Approximate Hardness, Rockwell C (b)		-	-	61	47	26	-			
Dial Reading, 30 N Scale		-	-	78-1/2	66	47	-			
Approximate Hardness, Rockwell C (b)		-	-	67	61	52-1/2	35			
Dial Reading, 45 N Scale		-	-	74	68	58	37			
Approximate Hardness, Rockwell B (b)		-	93	62	24	-	-			
Dial Reading, 15 T Scale		-	91	81	68	-	-			
Approximate Hardness, Rockwell B (b)		-	-	96	71	43	-			
Dial Reading 30 T Scale		-	-	79	64	45	-			
Approximate Hardness, Rockwell B (b)		-	-	-	90	70	32			
Dial Reading, 45 T Scale		-	-	-	62	43	4			

(a) For a given thickness, any hardness greater than that corresponding to that thickness can be tested. For a given hardness, material thicker than that hardness can be tested on the scale indicated.

(b) The approximate hardness numbers are for use in selecting a suitable scale and should not be used for hardness conversions.

TABLE XXVII. CORRECTIONS TO BE ADDED TO ROCKWELL SUPERFICIAL VALUES OBTAINED ON CYLINDRICAL SPECIMENS^(a) OF VARIOUS DIAMETERS⁽³²⁾

Dial Reading	Diameters of Cylindrical Specimens					
	1/8 in. (3.2 mm)	1/4 in. (6.4 mm)	3/8 in. (10 mm)	1/2 in. (13 mm)	3/4 in. (19 mm)	1 in. (25 mm)
Corrections to be Added to Rockwell Superficial 15 N, 30 N, and 45 N Values ^(b)						
20	6.0	3.0	2.0	1.5	1.5	1.5
25	5.5	3.0	2.0	1.5	1.5	1.0
30	5.5	3.0	2.0	1.5	1.0	1.0
35	5.0	2.5	2.0	1.5	1.0	1.0
40	4.5	2.5	1.5	1.5	1.0	1.0
45	4.0	2.0	1.5	1.0	1.0	1.0
50	3.5	2.0	1.5	1.0	1.0	0.5
55	3.5	2.0	1.5	1.0	0.5	0.5
60	3.0	1.5	1.0	1.0	0.5	0.5
65	2.5	1.5	1.0	0.5	0.5	0.5
70	2.0	1.0	1.0	0.5	0.5	0.5
75	1.5	1.0	0.5	0.5	0.5	0
80	1.0	0.5	0.5	0.5	0	0
85	0.5	0.5	0.5	0.5	0	0
90	0	0	0	0	0	0
Corrections to be Added to Rockwell Superficial 15 T, 30 T, and 45 T Values ^(b)						
20	13.0	9.0	6.0	4.5	3.0	2.0
30	11.5	7.5	5.0	3.5	2.5	2.0
40	10.0	6.5	4.5	3.5	2.5	2.0
50	8.5	5.5	4.0	3.0	2.0	1.5
60	6.5	4.5	3.0	2.5	1.5	1.5
70	5.0	3.5	2.5	2.0	1.0	1.0
80	3.0	2.0	1.5	1.5	1.0	0.5
90	1.5	1.0	1.0	0.5	0.5	0.5

(a) When testing cylindrical specimens, the accuracy of the test will be seriously affected by alignment of elevating screw, V-anvil, penetrators, surface finish, and the straightness of the cylinder.

(b) These corrections are approximate only and represent the averages, to the nearest 0.5 Rockwell superficial number, of numerous actual observations.

8.4.1 Sectioning and Mounting

After careful selection, samples can be sectioned on any type of a metallurgical cut-off machine. Allison C-120 SiC cut-off wheels are satisfactory for most materials. A continuous-rim diamond cut-off wheel, 0.040 inch, is used for sectioning coupons with ceramic coating. Coolant, usually tap water or water and cutting oil in a recirculating system, is used in all cutting operations. To avoid chipping the unsupported coating, it is sometimes advisable to mount the samples in epoxy resin before sectioning.

Samples may be mounted in Shell Epon Resin 815, using vacuum impregnation procedures to fill the pores. This is a liquid casting plastic which is easy to handle, has good adherence, hardness, and dimensional stability. Thermosetting powders such as Bakelite can be used, but mount size is limited, and Bakelite does not fill the pores as well as Epon. Two specimens are mounted together, positioning the coatings front to back, in the case of very hard specimens, or coating-to-coating, if the coating is soft or medium hard. The coatings themselves and the substrate material serve as backup to protect the coating.

8.4.2 Grinding

Samples are ground on a 10-inch disc grinder using 120-grit SiC grinding discs, to level the mount, using water as a coolant. Wet grinding continues on an 8-inch disc grinder, using successively finer SiC discs 180, 240, 400, and 600 grit. With each grit the mount is turned 90 degrees and the sample is ground until all scratches left by the preceding grit have been removed. After each step the mount is thoroughly washed with running water. Ultrasonic cleaning is never used for cleaning specimens of flame-sprayed coatings. Diamond laps 240, 400, and 600 grit are used instead of SiC discs to grind very hard materials such as ceramics.

8.4.3 Polishing

Preliminary polishing of soft metallic samples is done by hand on a low-speed (100 to 300 rpm) wheel using a medium-nap cloth charged with 3-micron diamond paste. Kerosene or lapping oil is used as a lubricant and polishing proceeds until all grinding scratches have been removed. Then the sample will be hand polished using 1-micron diamond on a medium-nap cloth. If better flatness and edge retention are desired, vibratory polishing can be used with 1-1/2-micron diamond on a silk cloth. Tungsten carbide coatings should not be polished on a vibratory polisher.

Diamond polishing causes excessive porosity in ceramics, cermets, and carbides. Overpolishing of tungsten carbide and hard-metal coatings on diamond create relief of the harder constituents

and pull-out of any loosely bonded constituents. Therefore, an alternative preliminary polishing technique is used for polishing some semihard to very hard materials. Grinding scratches are removed on a fast wheel (1750 rpm) covered with a low-nap cloth (nylon, silk, or lintless) and a slurry of 200 cc water, 4 grams chromium trioxide and 25 grams of "Linde A" polishing abrasive. If chromium trioxide preferentially attacks some phases, it can be reduced or eliminated from the slurry.

Final polish is obtained on a low-speed (100 to 300 rpm) wheel covered with a fine-nap cloth (Buehler microcloth or Gamal) with a slurry of 200 cc water, 4 grams chromium trioxide, and 20 grams ferric oxide. As in the preliminary polishing, chromium trioxide can be reduced or eliminated from the slurry to obtain the most satisfactory results. For some materials, 0.05 Gamma Alumina on a fine-nap cloth is used. Care should be taken not to polish too long, so as to avoid excessive relief of carbide and solid solution phases. When there are problems with galvanic attack due to the difference of potentials between brass or copper wheels, the substrate and the microconstituents in the coating, it can be eliminated by placing Saran Wrap between the wheel and the cloth.

8.4.4 Etching

Specimens are not etched before examining the coating for porosity or oxide contents. When other attributes are being examined, the coating is etched. The composition of the coating determines which etching technique is used: chemical, electrochemical, or vacuum cathodic etching.

Etching reveals the coating components by preferential attack or by staining the various phases. It is sometimes necessary to mask the substrate with tape before etching to eliminate galvanic attack between substrate and coating. Chemical etching is done by swabbing or by immersing the mount. Some coatings require electrolytic etching to reveal the structure. Some chemical etchants found useful for plasma-sprayed coatings are listed in Table XXVIII.

Structures of some coatings are revealed by the proper use of the chromium trioxide etch polish. Another method is to heat tint the surface. The color of the oxides formed on the polished surface relates to the composition of the carbide phases present.

Cathodic vacuum etching enjoys several advantages as compared with chemical etching techniques. It produces a surface free of stain and results in excellent relief delineation of the phases, which is desirable for studies with the electron microscope. Due to the time and expense, cathodic etching is used only for special evaluations.

TABLE XXVIII. SOME ETCHANTS FOUND USEFUL FOR METALLOGRAPHIC EXAMINATIONS OF PLASMA-SPRAYED COATINGS

Coating	Major Constituent	Etchants
Metco 450	Ni-4.5Al	50 cc lactic acid
Metco 404	Ni-20Al	10 cc HNO ₃
Metco 405	Ni-Al	50 drops HF
XP 1150	Ni-14.5Cr	Carapella's Etchant 5 g FeCl ₃ 2 cc HCl 98 cc ethyl alcohol Swab or immerse
XP 1151	Ni-25W	
Metco 439	Tungsten carbide	
Metco 438		
Metco 72F		
Metco 430		
Metco 501		
		Murakami's Reagent 10 g K ₃ Fe(CN ₄) 10 g KOH 100 cc H ₂ O Modified Murakami's 2.5 g NaOH 11.0 g K ₃ Fe(CN ₄) 500 cc H ₂ O Swab or immerse
Metco 54	Aluminum silicon	Keller's Reagent 95 cc H ₂ O 2.5 cc HNO ₃ 1.5 cc HCl 1.0 cc HF 60 cc glycerin 20 cc HNO ₃ 3 cc HF 100 cc H ₂ O 1 cc HF 50 cc HF 50 cc HNO ₃ 50 cc H ₂ O
Metco 43F	Nickel-chromium	100 cc H ₂ O
Metco 44		100 cc HCl
Metco 41C		20 g CuSO ₄
Metco 42C		90 cc HCl
Metco 43C		30 cc H ₂ O ₂
Metco 44C		Swab or immerse
Metco 45C		10% oxalic acid 6v DC electrolytic
Metco 91	Carbon steel	96 cc ethyl alcohol
Metco Sprasreel		4 cc HNO ₃ 96 cc ethyl alcohol 4 g picric acid Immerse

TABLE XXVIII. SOME ETCHANTS FOUND USEFUL FOR METALLOGRAPHIC EXAMINATIONS OF PLASMA-SPRAYED COATINGS (Continued)

Coating	Major Constituent	Etchants
Metco 439	Tungsten carbide	Murakami's
Metco 438		KOH-5% electrolytic
Metco 72F		

8.4.5 Metallographic Evaluation

Interpretation of microstructure and correlation of that information with the overall properties of the sprayed coating is a problem. In many preliminary metallographic investigations, qualitative information is all that is necessary.

On a production basis, the best method for determining whether a sprayed coating is acceptable is to compare its appearance with that of a standard material known to perform adequately in service.

For final evaluation of flame-sprayed coatings, quantitative information is almost always required. It is desirable to determine percentages of porosity, oxides, and other phases present in the flame-sprayed coating. Grid area point-count method and the Quantimet are the methods used for these determinations. Microhardness data can provide considerable information about the nature of a flame-sprayed coating. Difference in hardness is a valuable method for distinguishing between and identifying phases of a flame-sprayed coating. The type of hardness testing, whether Superficial Rockwell, Vickers, or Knoop, should be chosen according to the application. Microprobe and/or X-ray diffraction can be used to identify phases in flame-sprayed coatings during the research development stage of coating, but are not needed for production applications.

8.5 COATING POROSITY

Metallographic methods are widely used for judging the porosity of plasma-sprayed coatings. The quantitative approaches are based on the quantitative relationships between measurements on the two-dimensional plane of polish and the magnitudes of the microstructural features in three-dimensional materials. The applications and limitations of quantitative metallography have been described in detail by Underwood.⁽³³⁾ The underlying theories are well established. The principal sources of errors in estimating pore volumes are inadequately prepared metallographic specimens and taking too small a number of measurements. At the risk of oversimplification, the techniques used to determine the volume of voids, or discrete phases, in a microstructure can be briefly summarized.

The volume fraction occupied by a microstructural feature, such as a void or pore, is equal to the areal, linear, or point ratio of the selected feature as seen on random sections through the microstructure. Television-scanning equipment, such as the QuantimetTM instrument, is used for applications where the number of specimens examined makes automation justified. Linear ratios are commonly measured in a semiautomated fashion utilizing a Hurlbut Counter. The point-counting method is efficient and requires very little special equipment. The test points to be counted are those falling within the images of the microstructural feature (e.g., voids) being measured. The number of those points counted divided by the total number of available test points gives the volume fraction of the specimen occupied by voids. Ordinarily the array of available test points is provided by the intersections of a grid inserted in the eyepiece of a microscope. Alternatively, a clear plastic grid can be placed over micrographs and used for point counting. In either case, the grid spacing should be close to the spacing of the microstructural feature of interest (e.g., pores). Grid points falling on the boundary of a pore should be counted as one-half. Care must be taken in counting phases or voids not to overlook any or to count some twice.

Because point counting is tedious and special instruments for quantitative metallography are expensive, some organizations judge porosity by comparing the microstructures with standard photomicrographs. That method is simpler, quicker, and suitable for control and acceptance purposes. One large purchaser of plasma-sprayed components provides its vendors with micrographs illustrating its requirements for densities of coatings and for contents of oxides, inclusions, and unmelted particles. Depending on their purpose, comparisons are based on micrographs taken at magnifications ranging from 50 to 500X.

Although the two metallographic approaches just mentioned are recommended, some production shops use direct methods for measuring the porosity of plasma-sprayed coatings. The method suggested by a supplier several years ago consists, in general, of the following steps⁽²⁹⁾:

1. Prepare a solid cylindrical bar approximately 5-in. long X 7/8-in. in diameter; measure and record the diameter to 0.001 in.
2. Using the surface preparation methods and plasma-spray variables of interest and masks, apply a coating about 0.110-in. thick to the bar for a length of about 2.5 in.
3. Using the center holes, mount the coated bar in a lathe. Machine or grind the coated section to a thickness of 0.100 in., or 0.050 in. for self-fluxing coatings. Measure and record the diameter to 0.001 inch.

4. Cut specimens approximately 1.1-in. long from the coated and uncoated regions of the bar.
5. Grind both ends of both specimens flat and perpendicular to the central axis.
6. Weigh the specimens using an analytical balance to 0.001 grams (0.0000022 lb) and record the weight.
7. Calculate the volumes by the following equation:

$$\text{Volume} = \text{length} \times (\text{diameters})^2 \times (0.7854) \quad .$$

If that calculation is made in inches, divide the value by 0.081025 to obtain the volume in cm³.

8. Determine the density of the coating:

$$\frac{\text{Wt. of coated sample (g)} - \text{Wt. of uncoated sample (g)}}{\text{Vol. of coated sample (cm}^3\text{)} - \text{Vol. of uncoated sample (cm}^3\text{)}}$$

That "measured" density value can be divided by the "true" density of the coating material, determined as suggested by Section 8.2, page 95, or by another method, to express the density as a fraction of the "true" value.

The volume fraction of porosity can be obtained from "measured" and "true" density values in the following fashion:

$$\text{Void Volume Fraction} = \frac{(\text{"true" density} - \text{"measured" density})}{(\text{true density})}$$

8.6 SHOT PEENING TESTS

The efficacy of a shot peening operation depends on controlling the operation of the blasting operations and on their relationships to the work being peened. The effects of the peening operation can be measured, and thus controlled by adjusting the blasting variables, by tests on flat test strips. The evaluations are based on the fact that a thin metal strip blasted with shot while clamped to a solid block will be curved after removal from the fixture. The peened side of the curved strip will be convex; the degree of curvature is a measure of the residual stress developed in the strip and the severity of peening. The properties of the blast are the velocity, size, shape, density and hardness of the shot. The impingement angle, length of time of blasting, and shot flow rate are also important.

When peened substrates are to be plasma-arc sprayed, the just-mentioned variables should be controlled to give the appropriate peening intensity. The Society of Automotive Engineers has

adopted recommended practices for measuring the effects of shot peening that can be used for quality control. (34,35) The peening studies are made on SAE 1070 cold rolled steel test strips, uniformly hardened to Rockwell C 44 to 50 and flat to ± 0.001 inch.

Standard strip holders and gages, of the types shown in Figures 24 and 25, are used for the tests. During peening the test strip is mounted on the holder; after peening it is placed on the gage with the indicator stem bearing on the unpeened surface. The curvature of the strip is determined by measuring the height of the combined longitudinal and transverse arcs across standard chords. That arc height is obtained by measuring the displacement of a central point on the unpeened surface, to one-thousandth of an inch, from the four balls forming corners of a particular rectangle.

As indicated in Table XXIX, one of three different thicknesses of test strips is used for the evaluations. Strip A is recommended for peening test intensities that produce arc heights ranging from 0.006 to 0.024 inch. The relationships among arc heights for test strips A, C, and N after identical peening treatments are indicated in Table XXIX. Peening intensities are designated by the arc height in inches and the type of test strip employed, for example 0.004 A, 0.001 C, or 0.012 N.

TABLE XXIX. CORRELATION OF ALMEN PEENING INTENSITIES AS INDICATED BY ARC HEIGHTS OF STANDARD TEST STRIPS PEENED UNDER IDENTICAL BLASTING AND EXPOSURE CONDITIONS (34)

Almen Intensities for Test Strips Indicated, 0.001 inch(a)		
A Strip	C Strip	N Strip
2	-	6
4	-	12
6	1.8	18
8	2.4	24
10	3.0	-
15	4.5	-
20	6.0	-
25	7.5	-
30	9.0	-

(a) Test Strip Dimensions, inch:		
A	3.000 ± 0.015 x 0.745 to 0.750 x 0.051 ± 0.001	
N	3.000 ± 0.015 x 0.745 to 0.750 x 0.031 ± 0.001	
C	3.000 ± 0.015 x 0.745 to 0.750 x 0.094 ± 0.001	

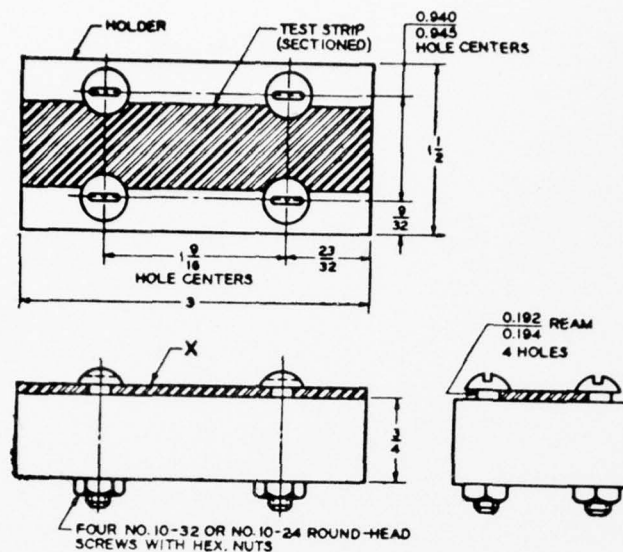


FIGURE 24. ASSEMBLED TEST STRIP AND HOLDER (34)

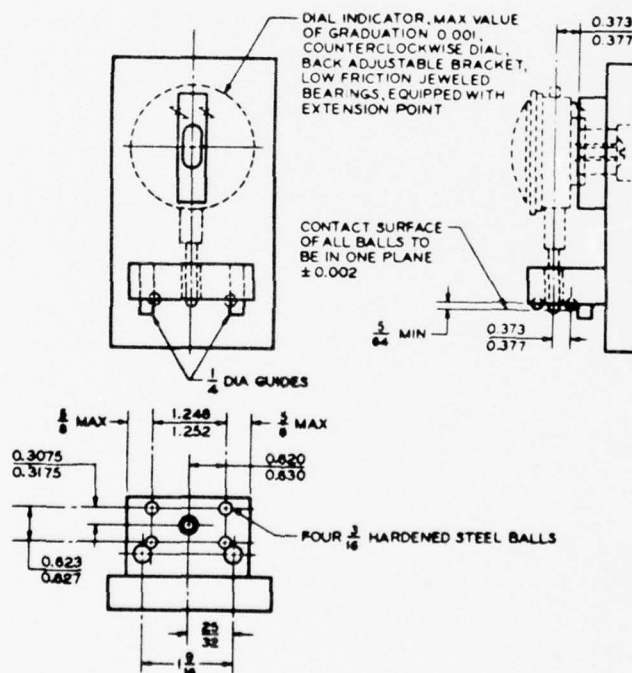


FIGURE 25. ALMEN GAGE NO. 2 (34)

The procedures for using the standard shot peening test strips, described in SAE J443, are briefly as follows⁽³⁵⁾:

1. Fasten the test strip tightly and centrally in the holder (Figure 24).
2. Expose the surface, X in Figure 24, to the peening conditions to be evaluated. Record the time used for blasting.
3. Remove the test strip from the holder and measure the arc height with the gage (Figure 25). The zero position of the gage must be checked frequently and, if necessary, adjusted.

The degree of coverage of shot peening can be estimated by the following procedure:

1. Polishing the strip to obtain a reflective surface.
2. Fastening the specimen to the test strip holder and peening under conditions identical to those to be used in production and for determining the Almen gage reading.
3. Removing the strip from the holder and placing it in the field of a metallurgical camera.
4. Using a piece of transparent paper as a ground glass and a magnification of 50X, trace the indented areas with a sharp pencil. The indented areas can be identified by the contrast with the polished surface.
5. Measure the area of the indentations within a known area using a planimeter. The ratio of the indented area to the total area is the percentage of coverage.

SECTION IX

9.

GLOSSARY

Abrasive: Material used for cleaning and/or surface roughening, usually sand, crushed chilled cast iron, crushed steel grit, aluminum oxide, silicon carbide, flint, garnet, or crushed slag.

Abrasive Blasting or Grit Blasting: The forceful direction of abrasive particles against the surface of workpieces or products to remove contaminants or to condition surfaces for subsequent operations.

Air Cooler: Work Piece Cooler, which see.

Anode: The electrode maintained at a positive electrical potential. In most plasma-torch designs this is the front electrode, constructed as a hollow nozzle and fabricated from copper.

Apparent Density or Density Ratio: The ratio of the measured density of an object to the absolute density of a perfectly solid material of the same composition, usually expressed as a percentage.

Arc: A luminous discharge of electrical current crossing the gap between two electrodes.

Arc Chamber: The confined space enclosing the anode and cathode in which the arc is struck.

Arc Gas: The gas introduced into the arc chamber and ionized by the arc to form a plasma.

Base Metal: Substrate, which see.

Base Material: Substrate, which see.

Berry Formation: A detrimental build-up of spray material on the gun nozzle and/or air cap during the spraying operation.

Blasting: A method of cleaning and/or surface roughening by a forcibly projected stream of sharp angular abrasive.

Bond: (1) To join securely. (2) A uniting force. (3) In thermal spraying, the junction between the material deposited and the substrate, or its strength.

Bond Cap: The term commonly used to describe a test specimen on which coating is applied for the purpose of testing bond strength.

Bond Coating or Bonding Coat: A thin, intermediate plasma-sprayed layer of a material (e.g., molybdenum) applied on the substrate to enhance the adherence of a subsequently sprayed coating.

Bond Strength: The force required to pull a coating free of a substrate.

Carrier Gas: In thermal spraying, the gas used to carry the powdered materials from the powder feeder or hopper to the gun.

Cathode: The electrode maintained at a negative electrical potential. Usually the rear electrode, conically shaped and fabricated from tungsten or thoriated tungsten.

Coating Density: The ratio of the determined density of the coating to the theoretical density of the material used in the coating process. Usually expressed as percent of theoretical density.

Coating Strength: A measure of the cohesive bond within a coating, as opposed to coating-to-substrate bond; the tensile strength of a coating.

Coating Stress: The stresses in a coating resulting from rapid quenching of molten or semimolten particles as they impact the substrate.

Coefficient of Thermal Expansion: The ratio of the change in length per degree of temperature to the length at 0 C or 0 F.

Composite Coating: A coating consisting of two or more layers of different spray materials.

Control Console: The instrumented unit from which the plasma torch is operated and operating parameters are monitored. Functions controlled and monitored are power level, stabilizing gas pressure and flow, powder-feed gas pressure and flow, and cooling water flow.

Controlled Atmosphere Chamber: An enclosure or cabinet filled with an inert gas in which plasma spraying or welding can be performed to minimize (or prevent) oxidation of the coating or substrate. The enclosure is usually fitted with viewing ports, glove ports to permit manipulations, and a small separate airlock for introducing or removing components without loss of atmosphere.

Density: The mass per unit volume of a material, usually expressed as grams/cubic centimeter or pounds/cubic inch.

Density Ratio: Apparent Density, which see.

Deposit: Spray Deposit, which see.

Deposit Efficiency: The ratio, by weight, of material deposited to material fed into the gun, measured by spraying on a large surface away from edges.

Deposition Rate: The speed with which material is deposited on a substrate, usually expressed in grams/minute or pounds/hour.

Dwell Time: The length of time the particles spend in the plasma stream.

Edge Effect: Loosening of the bond between the sprayed material and the base material at the edges, due to stresses set up in cooling.

Edge Loss: Material lost as overspray resulting from spraying near the edge of an object.

Elastic Modulus: The ratio of stress, within the proportional limit, to the corresponding strain. Young's Modulus is measured in tension or compression; the modulus of rigidity is measured by shear or torsion tests.

Electrode: An electrical conductor for leading current into or out of a medium. In arc and plasma spraying, the current-carrying components which support the arc.

Enclosure: For metal spraying, a chamber used for minimizing contamination. See Controlled Atmosphere Chamber.

Exhaust Booth: A mechanically ventilated, semi-enclosed area in which an air flow across the work area is used to remove fumes, gases, and overspray material during thermal spraying operations.

Feed Rate or Spray Rate: The quantity of material passed through the gun in a unit of time.

Fines: Those particles at the lower end of the specified mesh size.

Flame Spraying: A process in which materials are melted or softened in a heating zone and propelled in a molten or heat-softened (plastic) condition onto a target to form a coating. The term "flame" spraying is usually used when referring to a combustion-spraying process, as differentiated from "plasma" spraying or "plasma-flame" spraying.

Flow Meter: Device for indicating the rate of gas flow in a system.

Fretting: Surface damage resulting from relative motion between surfaces in contact under pressure, especially likely in a corrosive environment.

Fusion Spray: The process in which the coating is completely fused to the base metal, resulting in a metallurgically bonded, essentially void-free coating.

Galvanic Corrosion: Corrosion caused by the current between two dissimilar conductors in an electrolyte, or two similar conductors in dissimilar electrolytes. If the two dissimilar metals are in contact, the reaction is referred to as "couple action".

Gradated or Gradient Coating: A deposit which changes continuously but almost imperceptibly in composition from one surface to another, e.g., from 100 percent A at substrate to 100 percent B at top of the coating.

Graded Coating: A coating consisting of several successive layers of different materials; e.g., starting with 100 percent metal, followed by one or more layers of metal-ceramic mixtures, and finishing with 100 percent ceramic.

Grit: Abrasive, which see.

Grit Blasting: Abrasive Blasting, which see.

Gun: A term used to identify a thermal spraying device, especially the types used for depositing coatings by the plasma-arc or detonation-spray processes.

Inert Gas: A gas, such as helium, argon, or neon, which is stable and does not form reaction products with other materials.

Interface: A surface forming a common boundary between adjacent layers, usually the surface between the spray deposit and the substrate.

Ion: An atom or group of atoms (molecule) that carries a positive or negative charge as a result of having lost or gained one or more electrons.

Mask: A device for protecting a surface from the effects of blasting and/or coating. Masks are generally of two types: reusable or disposable.

Masking: The method of protecting the areas adjacent to the areas to be thermal sprayed or blasted to prevent adherence of a coating or surface roughening.

Matrix: The principal phase or aggregate, usually continuous, in which other constituents (such as oxides or carbides) are embedded.

Mechanical Bond: Adherence of a coating to a base material, accomplished mainly through mechanical interlocking with roughened surfaces.

Metallizing: Forming a metallic coating by spraying with molten metal or by vacuum deposition.

Metallurgical Bond: Adherence of a coating to the base material characterized by diffusion, alloying, or intermolecular or intergranular attraction at the interface between the sprayed particles and the base material; usually stronger than a mechanical bond.

Modulus of Rupture: Nominal stress at fracture in a bend test or a torsion test.

Open Circuit Voltage: The potential difference applied between the anode and cathode prior to initiating the arc.

Overspray: The excess spray material that is not deposited on the part being sprayed.

Parameter: A measurable constant or variable that is related to and affects other characteristics or variables describing a system or product.

Particle-Size Range: Classification of spray powders defined by an upper and lower size limit; e.g., -200 + 325 mesh: a quantity of powder, the largest particles of which will pass through a 200 mesh sieve and the smallest of which will not pass through a 325 mesh sieve.

Pass: A single progression of the thermal spray device across the surface of the substrate.

Plasma: An electrically neutral, highly ionized gas composed of ions, electrons, and neutral particles.

Plasma Flame: The zone of intense heat and light emanating from the orifice of the arc chamber resulting from energy liberated as the charged gas particles (ions) recombine.

Plasma-Forming Gas: The gas, in the plasma gun, which is heated to the high temperature plasma state by the electric arc.

Plasma Gases: Plasma Forming Gas, which see.

Plasma Gun: A device wherein a plasma is generated to provide a heat source for thermal spraying operations.

Plasma Spraying: Producing a coating by passing a material in particulate or powder form through a plasma flame and depositing the subsequently heat-softened particles onto a base material or substrate.

Plasma Torch or Plasma Gun: A device for producing a plasma flame, consisting of an arc chamber, an anode and a cathode, and equipped with cooling water, stabilizing gas, and powder-feed inlets and external power leads.

Powder Feeder: A mechanical device designed to introduce a metered and controlled flow of powder into the plasma-spray torch.

Powder-Feed Rate: The quantity of powder introduced into the arc per unit time; expressed in pounds/hour or grams/minute.

Powder-Feed Gas: The gas used to transport the powder from the feeder into the arc; usually an inert gas and most often argon.

ppm: Parts per million; 1 ppm is equal to 0.0001 percent.

Preheat: Heat applied to the base material prior to initiating the spray operation so as to avoid the presence of condensate on the substrate surface as the first particles are deposited or to minimize residual stresses.

Primary Gas: In thermal spraying, the gas constituting the major constituent of the arc gas fed to the gun to produce the plasma.

Quench Rate: The speed with which a sprayed particle cools upon striking the surface of the base material.

Rotary Roughening: A method of surface roughening wherein a revolving roughening tool is pressed against the surface being prepared, while either the work, or the tool, or both, move.

Relative Density: Apparent Density, which see.

Residual Stress: Stress present in a body that is free of external forces or thermal gradients.

Rough Threading: A method of surface roughening which consists of cutting threads with the sides and tops of the threads jagged and torn.

Seal Coat: Material applied to infiltrate the pores of a thermal spray deposit.

Secondary Gas: In thermal spraying, the gas constituting the minor constituent of the arc gas fed to the gun to produce the plasma.

Self-Bonding Materials: Those materials that exhibit the characteristics of forming a metallurgical bond with the substrate in the as-sprayed condition.

Self-Fluxing Alloys: Certain materials that "wet" the substrate and coalesce when heated to their melting point, without the addition of a fluxing agent.

sfm: Surface feet per minute; linear pass velocity in surface speed per minute.

Shadow Mask: Method of partially shielding an area during the spraying operation, thus permitting some overspray to produce a feathering at the coating edge.

Shielding Gas: A stream of inert gas directed at the substrate during spraying so as to envelop the plasma flame and substrate; intended to provide a barrier to the atmosphere in order to minimize oxidation.

Sieve Analysis: A method of determining particle size distribution, usually expressed as the weight percentage retained upon each of a series of standard screens of decreasing mesh size.

Spalling: Flaking or separation from the substrate of a sprayed coating.

Spray: A moving mass of dispersed liquid droplets or heat-softened particles.

Spray Angle: The angle of particle impingement, measured from the surface of the substrate and the axis of the spray nozzle.

Spray Deposit: A coating applied by any of the thermal spray methods.

Spray Distance: The distance maintained between the plasma-torch nozzle and the surface of the base material during spraying.

Spray Rate: Feed Rate, which see.

Spraying Sequence: The order in which different passes of similar or different materials are applied in a planned relationship, such as overlapping, superimposed, or at certain angles.

Stabilizing Gas: The arc gas, which is ionized to form the plasma. Introduced into the arc chamber tangentially, the relatively cold gas chills the outer surface of the arc stream tending to constrict the arc, raise its temperature, and force it out of the front anode nozzle in a steady, relatively unfluctuating stream.

Substrate: The material, workpiece, or substance on which the coating is deposited.

Substrate Preparation: The set of operations, including cleaning, degreasing, and roughening, applied to the base material prior to applying a coating; intended to insure an adequate bond to the coating.

Substrate Temperature: The temperature attained by the base material as the coating is applied. Proper control of the substrate temperature by phased spraying or by the application of external cooling will minimize stresses caused by thermal expansion differences.

Surface Preparation: The operations necessary to prepare a surface for thermal spraying.

Surface Roughening: A group of procedures for producing irregularities on the surface to be thermal sprayed. See Blasting, Rotary Roughening, Rough Threading, and Threading and Knurling.

Thermal Spray: Any coating process in which particles are heated to a molten or plastic state and propelled onto a substrate to form a coating; includes flame and plasma spraying using wire or powder processes.

Thermo Spray: A trade name for a particular flame-spray process. (Metco, Inc. powder flame-spray process.)

Threading and Knurling: A method of surface roughening wherein spiral threads are made and the tops of the threads are spread with a knurling tool.

Torch: Usually, a gas burner with feed lines for fuel and oxygen used to braze, cut, weld, or to heat material to be sprayed.

Transferred Arc: A plasma-spray process in which the workpiece is the anode for the plasma arc.

Traverse Speed: The lineal velocity at which the torch is passed across the substrate during the spraying operation.

Undercoat: A deposited coat of material which acts as a substrate for a subsequent thermal sprayed deposit. See Bond Coat.

Undercutting: A step in surface preparation involving removal of substrate material.

Water Wash: The forcing of exhaust air and fumes from a spray booth through water so that the vented air is free of thermal sprayed particles or fumes.

Wire Flame Spraying: Flame spraying metallic material fed to the torch or gun in wire or rod form.

Wire Flame Spray Gun: A flame spraying device utilizing an oxy-acetylene gas flame to provide the heat, and the metallic material to be sprayed in wire or rod form.

Wire Speed: The length of wire sprayed in a unit of time.

Wire Straightener: A fixture for taking the cast out of coiled wire to enable it to be easily fed into the gun.

Work Piece Cooler: A device used to direct an air blast onto a part being sprayed to prevent overheating of the part.

SECTION X

10.

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SECTION XI

11.

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SECTION XII

12.

COATING DATA SHEETS

12.1 BOND STRENGTHS

The average bond strengths for seven types of plasma-sprayed coatings deposited on five types of substrates are listed in Table XXX. The coatings were 0.015 to 0.022 inch (0.38 to 0.55 mm) thick. The oxide coatings were ground to 0.015 inch before testing, to insure flatness. The coatings were sprayed, in air, on bond-cap specimens. The strengths were determined by procedures conforming to the ASTM specifications for measuring adhesive and cohesive strengths.⁽³⁰⁾ The values are averages for six determinations on coatings deposited using controlled spraying parameters and surface preparation practices. Experience from these tests and other similar studies indicates that the reproducibility of values, from bond-strength tests considered reliable, is about ± 5 percent of the average value for six tests.

All of the substrates were degreased, grit blasted, cleaned with ultrasonic agitation in trichlorethylene, and washed in 200-proof alcohol. They were spray-coated within 1/2 hour of cleaning. The grit blasting was conducted at an air pressure of 60 psi with a nozzle-workpiece distance of 4 to 6 inches. The particle size of the alumina grit was described as being -60 + 40 mesh. As indicated in the table, those grit blasting conditions resulted in slightly different roughness values on substrates of different compositions.

The bond strength data in Table XXX were obtained on specimens spray coated using one particular type of plasma-gun equipment. However, many bond-strength tests were also made on coatings deposited with two other types of commercially available plasma-spray equipment. Those bond strengths agreed with the values listed in the table, within approximately 5 percent, when deposited under conditions described in Plasma Data Sheets.

12.2 PLASMA SPRAY VARIABLES

Parameters recommended for depositing seven types of coatings are described in the attached data sheets. The conditions are those found to be suitable by experiments with the three types of plasma-gun equipment mentioned in the sheets. The quality of the coatings was evaluated, using bond-strength tests, described above, microhardness tests, and metallographic examinations for porosity and for voids at the bond-substrate interface. It is believed that the conditions described in the sheets are suitable for producing high quality plasma-sprayed coatings. The plasma-spraying parameters are not appreciably affected by the substrate material.

TABLE XXX. BOND STRENGTHS OF PLASMA-SPRAYED COATINGS
DEPOSITED ON DIFFERENT SUBSTRATE MATERIALS (a)

Coating Material	Bond Strength, for Substrate Material Indicated, psi				
	Aluminum	Aluminum Bronze	Low Carbon Steel	316 Stainless Steel	K-Monel (b)
87TiO ₂ -13Al ₂ O ₃	3895	4175	4105	4165	4150
Cr ₂ O ₃ , 99%	5965	6220	6485	6450	6345
95.5Ni-4.5Al	4430	4725	4880	4885	4800
80Ni-20Cr	4310	4350	4455	4485	4541
Molybdenum, 99%	5075	5730	5920	5810	5745
Aluminum, 99.0+%	3965	4465	4405	4285	4270
Aluminum bronze	4085	4555	4670	4755	4715
Surface Roughness of Substrate, microinch AA					
	300	260	250	220	250

(a) Values are averages for six determinations. The plasma spray parameters are described in Data Sheets 1 through 7. Plasma sprayed coatings deposited according to methods described in Data Sheets 8 through 16 exhibited comparable bond strengths.

(b) The most recent designation for the material formerly known as K-Monel is K-500.

In all of the experiments summarized by data sheets 1 through 16, the following practices were held constant:

Substrate Surface	Flat
Spray Angle	90°
Preheating Temperature . .	150 F (65 C)
Coating Depth/Pass	0.002 inch (0.05 mm)
Coating Thickness	Up to 0.030 inch (0.75 mm)
Coating Speed, lineal, fpm	12.5
Powder-Carrier Gas	Argon
Carrier Gas Pressure . . .	50 psi
Substrates	1-in. D x 2.5 in.
Substrate Cooling Method .	Blast of dry air

If less-massive substrates than those used in these studies are to be plasma-arc coated, better substrate cooling techniques are desirable. Commercial devices marketed for that purpose are reputed to work satisfactorily.

Data sheets 17 through 25 describe spraying conditions used for applying plasma-arc coatings on the cylindrical rods mentioned in Section 2.1.1.2. The specifications of the powders used in that investigation are listed in Table XXI. In those studies the round substrates were rotated at 350 rpm, which resulted in a lineal traverse speed of 91 fpm. The conditions described in data sheets 17 through 21 were used to deposit coatings 0.030 inch (0.75 mm) thick in approximately 15 passes.

Data sheets 22 through 25 identify the plasma-spraying parameters used at Naval Ordnance Station, Louisville, to deposit coatings 0.022 inch (0.055 mm) thick on specimen cylinder rods. Nitrogen was used as the primary arc gas and as the powder-carrier gas in those processing operations. The coatings were considered comparable in quality to those produced using argon for arc and carrier gas and the conditions described in data sheets 17 through 21.

The data sheets identify the nozzles used for the plasma-arc deposition operations by manufacturer's component numbers. The following comparative information may be informative:

<u>Data Sheets</u>	<u>(Anode) Nozzle</u>	<u>Orifice ID, inch</u>	<u>Open-Circuit Voltage</u>
8-12	901065	0.313	80
13-16	S 1-3-F	0.312	80
1-7	GH	0.250	160
17-21	GH	0.250	160
22-25	G	0.249	160

FWB:ebk

PLASMA SPRAY DATA SHEET--1

Spray Material Alumina-Titania
Composition TiO_2 13%, Al_2O_3 87%
Size range, microns -53 + 15

Spray Gun

Type Metco 3MB
Nozzle GH
Powder port #2
Gun-to-work distance, inches 4
Gas
Primary Argon
Secondary Hydrogen
Flow, cfh
Primary 80
Secondary 25

Power

Amps 500
Volts 75
Kilowatt 37.5

Powder Feeder

Type Metco 3MP
Gear set Not applicable
Meter wheel S
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 24
Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 5

PLASMA SPRAY DATA SHEET--2

Spray Material Chromium Oxide
Composition Cr_2O_3 99.0%
Size range, microns -74 + 10

Spray Gun

Type Metco 3MB
Nozzle GH
Powder port #2
Gun-to-work distance, inches 3
Gas
 Primary Argon
 Secondary Hydrogen
Flow, cfh
 Primary 80
 Secondary 15

Power

Amps 500
Volts 65
Kilowatt 32.5

Powder Feeder

Type Metco 3MP
Gear set Not applicable
Meter wheel S
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 25
Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 7

PLASMA SPRAY DATA SHEET--3

Spray Material Nickel-Aluminum

Composition Nickel 95.5%, Aluminum 4.5%

Size range, microns -88 + 44

Spray Gun

Type Metco 3MB

Nozzle GH

Powder port #2

Gun-to-work distance, inches 5

Gas

Primary Argon

Secondary Hydrogen

Flow, cfh

Primary 80

Secondary 15

Power

Amps 500

Volts 65

Kilowatt 32.5

Powder Feeder

Type Metco 3MP

Gear set Not applicable

Meter wheel S

Feed Screw Not applicable

Vibration amplitude Not applicable

RPM 20

Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 9

PLASMA SPRAY DATA SHEET-- 4

Spray Material Nickel Chromium
Composition Nickel 80%, Chromium 20%
Size range, microns -105 + 44

Spray Gun

Type Metco 3MB
Nozzle GH
Powder port #2
Gun-to-work distance, inches 5
Gas

Primary	Argon
Secondary	Hydrogen
Flow, cfh	
Primary	100
Secondary	5

Power

Amps	400
Volts	60
Kilowatt	24

Powder Feeder

Type Metco 3MP
Gear set Not applicable
Meter wheel S
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 40
Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 19

PLASMA SPRAY DATA SHEET-- 5

Spray Material Molybdenum
Composition 99.0+%

Size range, microns	-74 + 44
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Spray Gun

Type Metco 3MB
Nozzle GH
Powder port #2
Gun-to-work distance, inches 4
Gas

Primary	Argon
Secondary	Hydrogen
Flow, cfh	
Primary	80
Secondary	15

Power

Amps	500
Volts	65
Kilowatt	32.5

Powder Feeder

Type Metco 3MP
Gear set Not applicable
Meter wheel S
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 15
Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 9

PLASMA SPRAY DATA SHEET-- 6

Spray Material Aluminum
Composition 99.0+
Size range, microns -53 + 44

Spray Gun
Type Metco 3MB
Nozzle GH
Powder port #2
Gun-to-work distance, inches 5
Gas
Primary Argon
Secondary Hydrogen
Flow, cfh
Primary 150
Secondary 5

Power
Amps 500
Volts 65
Kilowatt 32.5

Powder Feeder
Type Metco 3MP
Gear set Not applicable
Meter wheel S
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 32
Powder gas flow, cfh 37

Coating Data
Spray rate, lb/hr 6

PLASMA SPRAY DATA SHEET-- 7

Spray Material Aluminum Bronze

Composition Aluminum 9.5%, Iron 1.0%, Copper 89.5%

Size range, microns -105 +44

Spray Gun

Type Metco 3MB

Nozzle GH

Powder port #2

Gun-to-work distance, inches 5

Gas

Primary Argon

Secondary Hydrogen

Flow, cfh

Primary 150

Secondary 5

Power

Amps 500

Volts 65

Kilowatt 32.5

Powder Feeder

Type Metco 3MP

Gear set Not applicable

Meter wheel S

Feed Screw Not applicable

Vibration amplitude Not applicable

RPM 27

Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 14

PLASMA SPRAY DATA SHEET-- 8

Spray Material Alumina-Titania
Composition TiO_2 13%, Al_2O_3 97%
Size range, microns -53 + 15

Spray Gun

Type AVCO PG-100
Nozzle 901065
Powder port Not applicable
Gun-to-work distance, inches 3
Gas
 Primary Argon
 Secondary Not applicable
Flow, cfh
 Primary 70
 Secondary Not applicable

Power

Amps 600
Volts 33
Kilowatt 19.8

Powder Feeder

Type AVCO PF-200
Gear set Not applicable
Meter wheel Not applicable
Feed Screw 8 pitch
Vibration amplitude 50
RPM 300
Powder gas flow, cfh 7

Coating Data

Spray rate, lb/hr 5

PLASMA SPRAY DATA SHEET-- 9

Spray Material Chromium Oxide
Composition Cr_2O_3 99.0%
Size range, microns -74 + 10

Spray Gun

Type AVCO PG-100
Nozzle 901065
Powder port Not applicable
Gun-to-work distance, inches 3
Gas
Primary Argon
Secondary Not applicable
Flow, cfh
Primary 70
Secondary Not applicable

Power

Amps 600
Volts 33
Kilowatt 19.8

Powder Feeder

Type AVCO PF-200
Gear set Not applicable
Meter wheel Not applicable
Feed Screw 8 pitch
Vibration amplitude 50
RPM 300
Powder gas flow, cfh 7

Coating Data

Spray rate, lb/hr 5

PLASMA SPRAY DATA SHEET-- 10

Spray Material Nickel-Aluminum
Composition Aluminum 4.5%, Nickel 95.5%
Size range, microns -88 + 44

Spray Gun

Type AVCO PG-100
Nozzle 901065
Powder port Not applicable
Gun-to-work distance, inches 4
Gas
 Primary Argon
 Secondary Not applicable
Flow, cfh
 Primary 70
 Secondary Not applicable

Power

Amps 500
Volts 28
Kilowatt 14

Powder Feeder

Type AVCO PF-200
Gear set Not applicable
Meter wheel Not applicable
Feed Screw 8 pitch
Vibration amplitude 35
RPM 250
Powder gas flow, cfh 6

Coating Data

Spray rate, lb/hr 6

PLASMA SPRAY DATA SHEET--11

Spray Material Nickel Chromium
Composition Nickel 80%, Chromium 20%
Size range, microns -105 + 44

Spray Gun

Type AVCO PG-100
Nozzle 901065
Powder port Not applicable
Gun-to-work distance, inches 4
Gas
 Primary Argon
 Secondary Not applicable
Flow, cfh
 Primary 70
 Secondary Not applicable

Power

Amps 500
Volts 28
Kilowatt 14

Powder Feeder

Type AVCO PF-200
Gear set Not applicable
Meter wheel Not applicable
Feed Screw 8 pitch
Vibration amplitude 35
RPM 250
Powder gas flow, cfh 7

Coating Data

Spray rate, lb/hr 7

PLASMA SPRAY DATA SHEET--12

Spray Material Molybdenum
Composition 99.0%
Size range, microns -74 + 44

Spray Gun

Type AVCO PG-100
Nozzle 901065
Powder port Not applicable
Gun-to-work distance, inches 4
Gas Argon
 Primary Argon
 Secondary Not applicable
Flow, cfh
 Primary 70
 Secondary Not applicable

Power

Amps 500
Volts 28
Kilowatt 14

Powder Feeder

Type AVCO PF-200
Gear set Not applicable
Meter wheel Not applicable
Feed Screw 8 pitch
Vibration amplitude 35
RPM 250
Powder gas flow, cfh 7

Coating Data

Spray rate, lb/hr 9

PLASMA SPRAY DATA SHEET-- 13

Spray Material Alumina-Titania
Composition TiO_2 13%, Al_2O_3 87%
Size range, microns -53 + 15

Spray Gun

Type Plasmadyne SB 1B
Nozzle S1-3-F
Powder port Not applicable
Gun-to-work distance, inches 3
Gas
 Primary Argon
 Secondary Not applicable
Flow, cfh
 Primary 65
 Secondary Not applicable

Power

Amps 600
Volts 34
Kilowatt 20.4

Powder Feeder

Type Plasmadyne Roto-Feed 1000
Gear set A
Meter wheel Not applicable
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 80
Powder gas flow, cfh 12

Coating Data

Spray rate, lb/hr 5

PLASMA SPRAY DATA SHEET--14

Spray Material Chromium Oxide
Composition Cr_2O_3 99.0%
Size range, microns -74 + 10

Spray Gun

Type Plasmadyne SG 1B
Nozzle S1-3-F
Powder port Not applicable
Gun-to-work distance, inches 3
Gas
 Primary Argon
 Secondary Not applicable
Flow, cfh
 Primary 65
 Secondary Not applicable

Power

Amps 600
Volts 35
Kilowatt 21

Powder Feeder

Type Plasmadyne Roto-Feed 1000
Gear set A
Meter wheel Not applicable
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 80
Powder gas flow, cfh 12

Coating Data

Spray rate, lb/hr 6

PLASMA SPRAY DATA SHEET--15

Spray Material Nickel-Aluminum
Composition Aluminum 4.5%, Nickel 95.5%
Size range, microns -88 + 44

Spray Gun

Type Plasmadyne SG 1B
Nozzle S1-3-F
Powder port Not applicable
Gun-to-work distance, inches 4
Gas
 Primary Argon
 Secondary Not applicable
Flow, cfh
 Primary 60
 Secondary Not applicable

Power

Amps 500
Volts 30
Kilowatt 15

Powder Feeder

Type Plasmadyne Roto-Feed 1000
Gear set A
Meter wheel Not applicable
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 55
Powder gas flow, cfh 13

Coating Data

Spray rate, lb/hr 6

PLASMA SPRAY DATA SHEET-- 16

Spray Material Nickel Chromium
Composition Nickel 80%, Chromium 20%
Size range, microns -105 +44

Spray Gun
Type Plasmadyne SG 1B
Nozzle Sl-3-F
Powder port Not applicable
Gun-to-work distance, inches 4
Gas
 Primary Argon
 Secondary Not applicable
 Flow, cfh
 Primary 60
 Secondary Not applicable

Power
 Amps 450
 Volts 30
 Kilowatt 13.5

Powder Feeder
Type Plasmadyne Roto-Feed 1000
Gear set A
Meter wheel Not applicable
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 50
Powder gas flow, cfh 13

Coating Data
Spray rate, lb/hr 7

PLASMA SPRAY DATA SHEET--17

Spray Material Alumina-Titania
Composition Al_2O_3 87%, TiO_2 13%
Size range, microns -53 + 15

Spray Gun

Type Metco 3B
Nozzle GH
Powder port #2
Gun-to-work distance, inches 5
Gas

Primary Argon
Secondary Hydrogen
Flow, cfh
Primary 80
Secondary 25

Power

Amps 500
Volts 74
Kilowatt 37

Powder Feeder

Type 3MP
Gear set Not applicable
Meter wheel S
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 45
Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 3

PLASMA SPRAY DATA SHEET-- 18

Spray Material Chromium Oxide
Composition Cr_2O_3 99.0%
Size range, microns -140 + 10

Spray Gun

Type Metco 3MB
Nozzle GH
Powder port #2
Gun-to-work distance, inches 4
Gas
Primary Argon
Secondary Hydrogen
Flow, cfh
Primary 80
Secondary 15

Power

Amps 500
Volts 65
Kilowatt 32.5

Powder Feeder

Type 3MP
Gear set Not applicable
Meter wheel S
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 30
Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 3

PLASMA SPRAY DATA SHEET-- 19

Spray Material Nickel-Aluminum
Composition Nickel 95.5%, Aluminum 4.5%
Size range, microns -90 + 40

Spray Gun

Type Metco 3MB
Nozzle GH
Powder port #2
Gun-to-work distance, inches 5
Gas
 Primary Argon
 Secondary Hydrogen
Flow, cfh
 Primary 80
 Secondary 15

Power

Amps 500
Volts 65
Kilowatt 32.5

Powder Feeder

Type 3MP
Gear set Not applicable
Meter wheel S
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 40
Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 3

PLASMA SPRAY DATA SHEET-- 20

Spray Material Nickel Chromium
Composition Nickel 80%, Chromium 20%
Size range, microns -105 + 45

Spray Gun

Type Metco 3MB
Nozzle GH
Powder port #2
Gun-to-work distance, inches 5
Gas
 Primary Argon
 Secondary Hydrogen
Flow, cfh
 Primary 100
 Secondary 5

Power

Amps 400
Volts 60
Kilowatt 24

Powder Feeder

Type 3MP
Gear set Not applicable
Meter wheel S
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 40
Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 4

PLASMA SPRAY DATA SHEET-- 21

Spray Material Molybdenum
Composition 99.0%
Size range, microns -70 + 30

Spray Gun
Type Metco 3MB
Nozzle GH
Powder port #2
Gun-to-work distance, inches 5
Gas
 Primary Argon
 Secondary Hydrogen
 Flow, cfh
 Primary 80
 Secondary 15

Power
 Amps 500
 Volts 65
 Kilowatt 32.5

Powder Feeder
Type 3MP
Gear set Not applicable
Meter wheel S
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 45
Powder gas flow, cfh 37

Coating Data
 Spray rate, lb/hr 3-1/2

PLASMA SPRAY DATA SHEET-- 22

Spray Material Alumina-Titania

Composition Al_2O_3 87%, TiO_2 13%

Size range, microns -53 + 15

Spray Gun

Type Metco 3B

Nozzle G

Powder port #2

Gun-to-work distance, inches 5

Gas

Primary Nitrogen

Secondary Hydrogen

Flow, cfh

Primary 75

Secondary 15

Power

Amps 500

Volts 76

Kilowatt 38

Powder Feeder

Type 3MP

Gear set Not applicable

Meter wheel S

Feed Screw Not applicable

Vibration amplitude Not applicable

RPM 24

Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 3

PLASMA SPRAY DATA SHEET--23

Spray Material Chromium Oxide
Composition Cr₂O₃ 99.0%
Size range, microns -74 + 10

Spray Gun

Type Metco 3MB
Nozzle G
Powder port #2
Gun-to-work distance, inches 4
Gas

Primary	Nitrogen
Secondary	Hydrogen
Flow, cfh	
Primary	75
Secondary	15

Power

Amps 500
Volts 76
Kilowatt 38

Powder Feeder

Type 3MP
Gear set Not applicable
Meter wheel S
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 45
Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 5

PLASMA SPRAY DATA SHEET-- 24

Spray Material Nickel Aluminum
Composition Aluminum 4.5%, Nickel 95.5%
Size range, microns -88 + 44

Spray Gun

Type Metco 3MB
Nozzle G
Powder port #2
Gun-to-work distance, inches 5
Gas

Primary	Nitrogen
Secondary	Hydrogen
Flow, cfh	
Primary	80
Secondary	15

Power

Amps 500
Volts 65
Kilowatt 32.5

Powder Feeder

Type 3MP
Gear set Not applicable
Meter wheel S
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 15
Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 5

PLASMA SPRAY DATA SHEET--2

Spray Material Nickel Chromium
Composition Nickel 80%, Chromium 20%
Size range, microns -105 + 45

Spray Gun

Type Metco 3MB
Nozzle G
Powder port #1
Gun-to-work distance, inches 6
Gas

Primary	Nitrogen
Secondary	Hydrogen
Flow, cfh	
Primary	100
Secondary	15

Power

Amps 400
Volts 74
Kilowatt 29.6

Powder Feeder

Type 3MP
Gear set Not applicable
Meter wheel S
Feed Screw Not applicable
Vibration amplitude Not applicable
RPM 45
Powder gas flow, cfh 37

Coating Data

Spray rate, lb/hr 5

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER MT-043	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) A PLASMA FLAME SPRAY HANDBOOK.		5. TYPE OF REPORT & PERIOD COVERED Final Report
7. AUTHOR(s) T. J. Roseberry and F. W. Boulger (Battelle Memorial Institute)		6. PERFORMING ORG. REPORT NUMBER N-00197-73-C-4030
9. PERFORMING ORGANIZATION NAME AND ADDRESS Battelle Columbus Laboratories 505 King Avenue Columbus, Ohio 43201		8. CONTRACT OR GRANT NUMBER(s) WR-3-5961
11. CONTROLLING OFFICE NAME AND ADDRESS Naval Sea Systems Command Department of the Navy, Manufacturing Technology Br. (SEA-0354), Washington, D.C. 20362		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) Naval Ordnance Station (CC 85) Southside Drive Louisville, Ky. 40214		12. REPORT DATE March 1977
		13. NUMBER OF PAGES 164
		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Approved for Public Release - Distribution Unlimited		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Plasma, Metal Spraying, Flame Spraying, Thermal Spraying, Handbook, Powder Spraying, Spraying, Buildup, Repairing, Overhaul		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The Plasma Spraying process is a versatile fabrication technique used to apply a wide range of coatings on various workpiece materials. Coatings are applied to restore or attain desired dimensions, to provide electrical or thermal shielding (or conduction), or to improve the resistance to abrasion, corrosion, or high temperatures. Success in meeting intended goals by Plasma Spraying depends on selecting an appropriate coating material and using cleaning and (Continued on reverse)		

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spraying techniques suitable for the materials involved. This report provides useful, up-to-date information about Plasma Spraying both metallic and non-metallic coatings on steel and bronze. It discusses the principles and details the procedures suitable for producing high-quality coatings destined for severe service applications of interest to the U. S. Navy.

This handbook should be considered a guide, not a bible, for planners, designers, and production personnel responsible for choosing, applying, and evaluating plasma-arc sprayed coatings. Limitations on space and reliable information, and the variety of types and applications of coatings, prevented covering some aspects of plasma spraying in detail. For such reasons, differences in the types of plasma-spray systems used successfully, limiting coating thickness, and effects of specific part geometries are not covered comprehensively. Most of the information in the handbook is directed to engineers overseeing and developing expertise in plasma-arc spraying.

The handbook is based on experience, published information, and experimental data generated on this program. The experimental work was conducted with seven types of coatings deposited on the following types of workpiece materials:

Aluminum
Aluminum bronze
K-Monel

Low-carbon steel
316 stainless steel